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TECHNICAL REPORT
FD-26

OBJECTIVE TESTS FOR USE IN THE TECHNOLOGY OF COMPRESSED FOODS

by

E. R. Morris

MIDWEST RESEARCH INSTITUTE
Kansas City, Missouri

Contract No. DA 19-129-AMC-130(N)

September 1965

U. S. Army Materiel Command
U. S. ARMY NATICK LABORATORIES
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FOREWORD

This investigation was undertaken as a part of a comprehensive program directed to the development of food packets to be carried by the combat soldier during periods in which resupply is impracticable. The necessity for reducing the volume as well as the weight of component foods together with the packaging efficiency from geometrically regular modules, point to the advantages of compressing or otherwise compacting dry foods into dense bars. Developments in bar technology, such as the use of freeze dried foods, special compression procedures and use of high caloric binding agents favorable to desirable physical and organoleptic properties, point to the suitability of such bars not only for combat food packets but also for other operational rations. This investigation was undertaken in recognition of the growing significance of novel types of food bars in operational rations and the many problems attending the development, fabrication, testing, and ultimate procurement of these bars. It is highly desirable that the essential characteristics of compacted food bars be identified and described by values obtained from objective measurements. Only through such measurements can precise and reproducible values be assigned to significant properties, for example, hardness, stickiness, cohesiveness, and dispersibility in water. Objective measurements will also provide a sound basis for definitive requirements in specifications and for improved inspection and quality control procedures. It is the primary objective of this investigation to identify objective methods suitable for describing significant physical, chemical and microbiological characteristics of compacted food bars of varying types and compositions.

This report describes work performed under contract DA19-129-AMC-130 in the Food Technology and Nutrition Section of the Biological Sciences Division at the Midwest Research Institute, 425 Volker Boulevard, Kansas City 10, Missouri. Dr. Eugene R. Morris served as Official Investigator with Glenn Chaplin, Emil Werly, Barbara Kenagy and Audra Calhoon as Collaborators.

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ABSTRACT

Objective methods are described for determining specific physical, chemical and microbiological properties of compressed food bars. The suitability of these methods was demonstrated by application to fresh and aged (3 months at 100°F) bars prepared from meat, fruit, cereals, vegetables, and dairy products and which represented broad concentration ranges of moisture, fat, protein, carbohydrate, and common approved chemical additives.

OBJECTIVE TESTS FOR USE IN THE TECHNOLOGY OF COMPRESSED FOODS

SUMMARY

The objective of Midwest Research Institute Project No. 2709-B, "Objective Tests for Use in Technology of Compressed Foods," was to develop analytical procedures by which the physical and chemical characteristics and the microbiology of compressed-dehydrated food bars may be quantitatively described. The work was performed under Contract No. DA-19-129-AMC-130(N) for the U. S. Army Natick Laboratories, Natick, Massachusetts.

Objective analytical procedures were developed for quantitatively describing the following characteristics of compressed dehydrated food bars:

Physical characteristics:

1. Density
2. Porosity
3. Shear and hardness
4. Impact resistance
5. Dimensional stability
6. Cohesiveness
7. Stickiness
8. Dispersibility in water
9. Water activity at 25°C
10. Elasticity
11. Permeability to gas

Chemical characteristics:

1. Moisture content

2. Free lipid and bound lipid content

3. Reducing sugars

4. pH

5. Water-soluble fraction

6. Equilibrium relative humidity curve and rate of equilibration to 40 per cent R. H.

7. Rate and amount of gaseous oxygen uptake

8. Changes associated with oxygen uptake

9. Changes associated with browning reaction

Microbiology:

1. Total aerobic plate count

2. Coliform plate count

3. Anaerobic spore count

These methods were used to quantitatively evaluate the foregoing characteristics for compressed food bars made from: (1) freeze-dried cooked ground beef; (2) air-dried dates; (3) freeze-dried cooked peas; (4) cereal; (5) nonfat milk solids; and (6) potato chips. The beef, date, pea and cereal bars were prepared at two different moisture levels to determine the effect of moisture on these characteristics. The effects of additives to the beef bars of 3 per cent sorbitol and 2 per cent mono- and diglycerides and acetyl glyceride coatings on the date bars were also determined.

Methods for preparing the above food bars are described.

The above characteristics were checked, first on the freshly prepared bars, and then after three months' storage, at four different storage conditions: (1) at 100°F in a sealed can with pO_2 equal to 135 - 145 mm. Hg; (2) at 100°F in a sealed can with pO_2 below 1 mm. Hg; (3) in a sealed can with repeated temperature cycling between 0 and 40°F, three cycles per week; and (4) at 70°F in air at relative humidity of 40 per cent.

Any limitations or deficiencies found in the methods are pointed out. In those instances in which the particular method was found to be inaccurate, other methods are suggested. The applicability of some of the characteristics to the technology of compressed foods is discussed, and suggestions are made for future work.

I. INTRODUCTION

Compressed food bars made from dehydrated food items provide for the Armed Forces the advantages of greater convenience and stability and a higher concentration of energy and nutrients than conventional canned foods. New products, such as the compressed food bars, require the development of suitable specifications for their production. These specifications, in turn, require methods of evaluation. The objective of Contract No. DA 19-129-AMC-130(N) has been to develop objective tests for use in the technology of compressed food bars.

Methods were devised for the objective measurement of specified physical and chemical characteristics, and of the microbiological characteristics of several different compressed food bars. These methods were evaluated by testing freshly prepared compressed food bars. In addition, measurements were made on samples of the bars which had been stored under several different storage conditions to determine the effects of storage on the various parameters studied.

The next section of this report describes the methods used for measuring the various physical and chemical characteristics and the microbiological population of the compressed food bars. The results of measurements of the freshly prepared bars are included in this section. The third section reports the results of measurements of these characteristics after the food bars had been stored under four different conditions for three months. The third section is followed by a brief description of the preparation of the various food bars. A section on conclusions and recommendations is included.

II. METHODS USED AND RESULTS OF OBJECTIVE MEASUREMENTS OF COMPRESSED FOOD BAR CHARACTERISTICS

The methods described in this section utilized, insofar as possible, procedures recognized by the Association of Official Agricultural Chemists, ASTM procedures, or procedures published in textbooks on foods. A few were found to be not applicable for the specific purpose desired, and the reasons will be pointed out in the discussion of each individual method. Included in the discussion of each test and the method used are the results of tests made on freshly prepared compressed food bars. The figures reported represent the results obtained from at least five bars of each kind or aliquots of five bars. The terms "high" and/or "low moisture" are used as descriptive terms throughout this report, and the reader is referred to the section on moisture determination in this chapter (see p. 25) for the actual moisture content of the various types of food bars.

A. Physical Characteristics

1. Density

a. Method: The bulk density of the compressed food bars was determined after the method described in ASTM[1] for determining the density of urethane foam. The dimensions of an intact bar were measured by means of calipers capable of measuring to 0.001 in., and the mass was determined on an analytical balance to the nearest 0.0001 g. The bulk volume in cubic centimeters was then calculated, as was the bulk density in grams per cubic centimeter. The surface of the bars was sufficiently regular and the bars were solid enough that the foot area (approximately 1 x 1/16 in.) of the calipers did not require augmentation.

b. Results: The bulk densities thus determined for the different food bars are shown in Table I. The values ranged from about 0.5 g/cc for the beef bars to about 1.2 g/cc for the date bars.

Table I also shows "apparent true density" values. True density is calculated by dividing the mass by the true volume of solids. The true volume of solids is obtained by subtracting the pore volume from the bulk volume. Our early methods of determining pore volume subsequently were found to be inaccurate. Therefore, our results are reported as "apparent true density" rather than "true density." Those bars which were rather porous - beef, peas, cereal, and nonfat milk solids - had apparent true densities which were 130 - 140 per cent of the bulk density. Because date bars had a low porosity, the apparent true density of solids was essentially the same as the bulk density.

No difficulties were experienced in determining the bulk density by the method described. However, the accuracy of the method used for determining porosity governs the accuracy with which the true density may be determined.

TABLE I

DENSITY OF COMPRESSED FOOD BARS

Food Bar	Bulk Density g/cc	Apparent True Density of Solids* g/cc	Apparent True Density of Solids* lb/ft^3
Beef, low moisture	0.47 (0.46-0.48)**	0.71	44.3
Beef, high moisture	0.45 (0.39-0.48)	0.67	41.8
Beef, w/sorbitol	0.47 (0.45-0.50)	0.72	45.0
Beef, w/mono- diglycerides	0.50 (0.48-0.52)	0.76	47.4
Dates, low moisture	1.21 (1.15-1.25)	1.25	78.0
Dates, high moisture	1.17 (1.08-1.21)	1.23	76.8
Dates, w/acetyl glyceride coating	1.18 (1.16-1.21)	1.20	74.9
Peas, low moisture	0.68 (0.66-0.70)	1.04	64.9
Peas, high moisture	0.67 (0.64-0.68)	1.00	62.4
Cereal, low moisture	0.90 (0.84-0.91)	1.19	74.3
Cereal, high moisture	0.88 (0.86-0.90)	1.16	
Nonfat milk solids	1.05 (1.04-1.05)	1.33	83.0
Potato chips	0.70 (0.67-0.72)	0.89	55.6

* Volume of solids determined by subtracting the pore volume determined by use of the McLeod Porosimeter from the bulk volume of the food bars.

** Range of values shown in parentheses.

2. Porosity

a. Method: The porosity or pore volume was determined by a modification of the ASTM method for determining porosity of vitrified ceramic materials.2/ This method involves placing the sample in the air-tight chamber of a McLeod type porosimeter2,3/ which is then emptied of air by filling the closed chamber with a liquid. The chamber is then evacuated to cause withdrawal of entrapped air from the sample. The volume of entrapped air thus extracted is measured by means of a gas burette. The volume of solids is calculated by subtracting the amount of gas extracted from the bulk volume. We modified the standard method by substituting mercury for water as the confining liquid.

b. Results: This method was found to be inaccurate when the volume of solids thus determined was compared with measurements made with an air comparison pycnometer.* The values for porosity of the food bars shown in Table II were determined by the modified ASTM procedure. The air comparison pycnometer was only used during the latter stages of the project. Using the air comparison pycnometer, average pore volumes of 63 per cent and 25 per cent, respectively, were obtained for beef and nonfat milk solids bars.

The source of error using the McLeod porosimeter has not been determined. The air comparison pycnometer is more accurate and easier to use so we recommend its use to determine true volume of solids.

Washburn and Bunting3/ discuss an apparatus and method for determining the pore volume of whole bricks based on the change in pressure of an evacuated system of known volume when the air in the brick is allowed to expand into the evacuated space. A rather inexpensive apparatus can probably be constructed, and this method adopted for measuring the pore volume of compressed food bars.

3. Shear and Hardness

a. Method (shear): A shear blade was constructed and used in a Warner-Bratzler meat tenderness tester to simulate the action of incisors shearing a food block. The blade was made from iron sheet,

* Air comparison pycnometer, manufactured by Beckman Instruments, Inc., Fullerton, California.

TABLE II

POROSITY MEASUREMENTS OF COMPRESSED FOOD BARS

(pore volume as per cent of bulk volume)

Food Bar	% Pore Volume Avg. Value	Range
Beef, low moisture	33	32-34
Beef, high moisture	32	28-34
Beef, w/sorbitol	34	34-36
Beef, w/mono-, diglycerides	35	34-35
Dates, low moisture	3	2-3
Dates, high moisture	5	4-5
Dates, w/acetyl glyceride coating	5	4-5
Peas, low moisture	35	34-36
Peas, high moisture	33	32-34
Cereal, low moisture	24	24-25
Cereal, high moisture	24	24-25
Nonfat milk solids	21	21-22
Potato chips	21	21-22

2 x 3 x 0.006 in. A 45° bevel was milled across the 2-in. face of the blade, and the sharp edge of the bevel was rounded off on a grinding wheel over approximately one-third the thickness of the blade and extending back about 0.002 in. from the original edge of the blade in the direction of the opposite surface (Fig. 1). The blade was mounted in a vertical position on a yoke which, in turn, was attached to the measuring device of a Warner-Bratzler meat tenderness tester. To determine shear on a food bar, the bar was placed on edge against the mounting block of the tester. The mounting block was advanced mechanically and pressed the food bar against the shear blade. The blade and bars were mounted in such a manner that the bar was cut across its 1-1/4 in. sq. face. The force necessary for the blade to shear through the block was registered on the measuring device of the tester (Hansen model 60 dairy scale). The shearing action thus obtained is not the same as if a sharp knife edge were pressed into the bar. The pressure at which the bar divided was called the end point. The end point was very sharp for most of the food bars; they parted suddenly and sharply. In the case of the date bars, the blade gradually sheared through the bar as pressure was applied, and the end point was taken as the pressure registered when the blade had been pushed entirely through the bar. The beef bars were not soft but were fragile, and a like end point was used for them.

b. Results (shear): Shear values obtained on the different compressed food bars are tabulated in Table III. The beef and cereal bars were rather crumbly and had shear values of less than 10 lb. The high moisture date bars were soft and shear values were low, but the low moisture date bars had shear values of 25 to 30 lb. The pea and nonfat milk solids bars were hard to bite and the shear values were high, about 40 lb.

c. Method (hardness): A type A Shore durometer was first used to determine hardness, but it proved to be unsatisfactory. The penetrating pin was too small in diameter to be used on some of the more porous materials, and small particles and sticky material from the surface of the bars caused the pin to hang.

Consequently we designed a penetration device to be used in the Warner-Bratzler tester. Figure 2 is a diagram of this device which consisted of four pins, 0.11 in. dia., set into an aluminum alloy block. The device was set against a yoke attached to the measuring device of the Warner-Bratzler tester with the pins pointed toward the food bar. The food bar was set on edge against the mounting block of the tester.

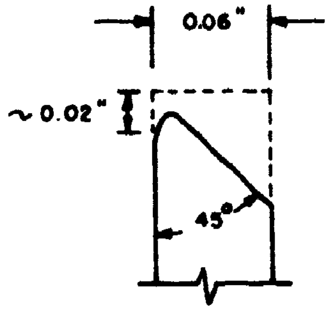

Fig. 1 - Cross-sectional Diagram of the Edge of the Shear Blade

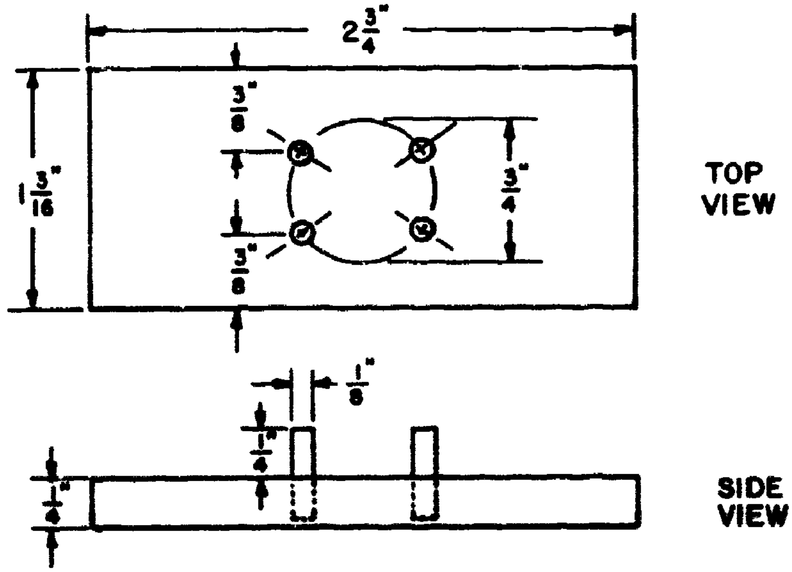

Fig. 2 - Diagram of the Device Used with the Warner-Bratzler Tester for the Hardness Test

TABLE III

HARDNESS AND SHEAR MEASUREMENTS OF COMPRESSED FOOD BARS

Food Bar	Hardness, Lb. Avg.	Hardness, Lb. Range	Shear, Lb. Avg.	Shear, Lb. Range
Beef, low moisture	5	2-6	3	2-4
Beef, high moisture	5	4-7	3	2-6
Beef, w/sorbitol	4	4-5	2	1-2
Beef, w/mono-, diglycerides	5	4-7	2	1-3
Dates, low moisture	23	22-25	27	25-30
Dates, high moisture	4	3-4	5	4-6
Dates, w/acetyl glyceride coating	5	3-6	6	5-8
Peas, low moisture	61	59-70	41	35-45
Peas, high moisture	52	42-63	38	22-63
Cereal, low moisture	16	13-18	6	4-8
Cereal, high moisture	19	14-22	8	7-9
Nonfat milk solids	> 70	> 70	42	34-48
Potato chips	14	10-16	10	8-12

As the mounting block was mechanically advanced, the food bar was pressed against the projecting pins. The end point was taken as the pressure at which complete penetration occurred. As was the case with the shear test, the end point for the hard bars was usually sharper than for the soft bars.

d. Results (hardness): Results of the hardness test, Table III, exhibited the same general relations among the different types of compressed food bars as did the shear test. In general, the hardness values were higher than the shear values.

Research might profitably be performed to determine the optimum configuration, diameter and number of pins for a device such as the one used for the hardness test. A similar type device which has a head with a larger number of prongs is commercially used for testing the texture of apples.*

4. Impact Resistance

a. Method: A Gardner impact tester[4/] equipped with a 1-lb. weight was used to test impact resistance. The food bar was placed on a flat surface beneath the guide of the tester. The weight was allowed to drop onto the surface of the bar. If the bar did not shatter, the drop was repeated from a greater height until it shattered. A fresh bar was then placed under the weight and the weight raised to the maximum height attained with the first bar. If the bar did not shatter, a fresh bar was substituted and the height increased by increments of 1 in. The procedure was repeated until a fresh test bar shattered on the first attempt. Thus, the figure determined for impact resistance represents the inch-pounds of force necessary to produce shattering. The bar was not considered to have shattered unless pieces were actually dislodged from it.

b. Results: The values obtained for impact resistance of the test food bars are summarized in Table IV. The beef bars had a very low impact resistance. By the above definition of shattering, the date bars did not shatter, even though the weight dented the bar. The pea bars had the highest impact resistance, while the cereal and potato chip bars had a relatively low value. Even though the nonfat milk solids bars were quite hard, they had a low impact resistance.

* The texturemeter, Seifert Manufacturing Company, St. Nazian, Wisconsin.

TABLE IV

IMPACT RESISTANCE MEASUREMENTS ON COMPRESSED FOOD BARS

Food Bar	In.-Lb. of Force to Cause Shattering	
	Avg. Value	Range
Beef, low moisture	0.5	-
Beef, high moisture	0.5	-
Beef, w/sorbitol	0.5	-
Beef, w/mono- diglycerides	0.5	-
Dates, low moisture	No shattering	
Dates, high moisture	No shattering	
Dates, w/acetyl glyceride coating	No shattering	
Peas, low moisture	9.0	-
Peas, high moisture	12.0	-
Cereal, low moisture	1.5	-
Cereal, high moisture	1.5	-
Nonfat milk solids	0.6	0.5-0.8
Potato chips	1.5	-

5. Dimensional Stability

a. Method: Dimensional stability was tested by placing the test food bar under a compressive force of 5 psi for 24 hr. Each test bar was sealed in a foil laminate packet so there would be no change in moisture content over the 24-hr. period. These packets were large enough that expansion of the bar was not restricted. Metal bars which weighed 7.8 ± 0.05 lb. (equivalent to 5 psi on the 1-1/4 in. sq. bars) were then placed on the bar for 24 hr. The bars were supported in a guide which held them upright but allowed the weight to slide if the test food bar collapsed. The linear measurements before and after the test period were determined and the dimensional stability reported as per cent change from the original dimensions. The test was conducted at two temperatures, 40° and 100°F. The test bar was allowed to come to the test temperature before the weight was applied.

b. Results: The results obtained on the test compressed food bars are shown in Table V. None of the bars underwent more than an 8 per cent change in either thickness or lateral dimensions when the test temperature was 40°F. The beef, pea, low moisture cereal, and nonfat milk solids bars, especially the last, showed good dimensional stability at 100°F, while the high moisture cereal and potato chip bars underwent some deformation. However, the date bars underwent considerable deformation, from 50 per cent to over 100 per cent change in lateral dimensions and an average of about 50 per cent change in thickness.

The importance of allowing the bars to reach temperature equilibrium is illustrated by the date bars. When the high moisture date bars were placed in the 40°F test chamber and the weight applied while they were still at room temperature, the deformation was about 75 per cent of that attained in the 100°F test chamber. However, as shown in Table V, the high moisture date bars were quite stiff and resistant to dimensional change under 5 psi when allowed to come to temperature equilibrium at 40°F before the weight was applied.

6. Cohesiveness

a. Method: Szczesniak[5] has defined "cohesiveness" as the strength of the internal bonds making up the body of the product. The procedure developed for measuring cohesiveness of the compressed food bars tested the strength of the internal bonding by pulling the bar

TABLE V

DIMENSIONAL STABILITY OF COMPRESSED FOOD BARS
AT 40° AND 100°F UNDER 5 psi

(per cent change in dimensions in 24 hr.*)

Food Bar	Temp. °F	% Lateral Change Avg.	% Lateral Change Range	% Thickness Change Avg.	% Thickness Change Range
Beef, low moisture	40	2	1-3	4	3-5
	100	3	2-4	11	8-15
Beef, high moisture	40	< 1	NC**1	2	1-4
	100	< 1	NC - 1	5	4-5
Beef, w/sorbitol	40	< 1	NC - 1	4	3-4
	100	1	NC - 4	5	4-6
Beef, w/mono- diglycerides	40	< 1	NC - 1	2	2-3
	100	< 1	NC - 1	5	4-6
Dates, low moisture	40	4	2-6	5	4-6
	100	49	44-54	34	33-34
Dates, high moisture	40	4	4-5	8	6-9
	100	147	112-188	59	51-64
Dates, w/acetyl glyceride coating	40	2	NC - 5	4	2-5
	1[illegible]	157	130-189	64	60-67
Peas, low moisture	40	< 1	NC - 1	< 1	NC-1
	100	< 1	NC - 1	1	1-2
Peas, high moisture	40	< 1	NC - 1	2	1-3
	100	< 1	NC - 2	8	5-11
Cereal, low moisture	40	< 1	NC - 1	2	1-3
	100	< 1	NC - 1	1	1-2
Cereal, high moisture	40	< 1	NC - 1	2	1-3
	100	14	11-18	16	13-18
Nonfat milk solids	40	NC	-	NC	-
	100	NC	-	NC	-
Potato chips	40	< 1	NC - 1	2	1-3
	100	7	7-8	12	10-12

* Test bars were brought to temperature equilibrium with test chamber before weight was applied.

** NC = no change.

apart. Holes were drilled in diagonally opposite corners of the food bar to be tested, approximately 1/4 in. from each edge. A mold was made to enable these holes to be placed in the same position in each corner and in each bar. The bar was then mounted on the Warner-Bratzler tester by slipping the bar over pins secured to the mounting block and the measuring device of the tester. As the mounting block of the tester was mechanically advanced, a pulling force was applied at opposite corners. The force being applied when the bar broke apart was read from the tester scale.

b. Results: The cohesiveness of the beef, cereal and potato chip bars was so low that the mounting holes could not be drilled without the bars breaking or crumbling. Holes could be drilled in the pea bars, but the high moisture pea bars pulled apart before a measurable force was applied (see Table VI). The date bars could be mounted, but only the low moisture group parted after a measurable force had been applied. Twelve pounds of force was required to pull the bars of nonfat milk solids apart.

7. Stickiness

a. Method: A small disc, 0.8 in. in diameter and 0.025 in. thick, was made from aluminum sheet and a hook attached to hang it from the arm of a Du Nouy tensiometer. The tensiometer, with the disc attached, was calibrated with analytical weights and found to exert a force of 7.9 dynes/dial division or, assuming the disc was attached to another surface, 15.2 dynes/sq in. of surface per dial division. Bars were equilibrated to atmospheres of approximately 40 per cent and 80 per cent R.H. before testing for stickiness. The disc was set on the flat surface of the test bar under a weight of 20 g. for 2 min., then the force necessary to pull the disc away was measured on the tensiometer.

b. Results: Only date bars exposed two weeks to 80 per cent R.H. had any measurable stickiness by this method. The stickiness was greater than the instrument could measure. The maximum measurable force exerted by the tensiometer with the disc was equivalent to 1,670 dynes/sq in. The date bars were the only ones which exhibited any stickiness when touched.

TABLE VI

COHESIVENESS MEASUREMENTS IN COMPRESSED FOOD BARS

(pounds force to pull the bars apart)

Food Bar	Cohesiveness Avg. Value	Cohesiveness Range
Beef, low moisture[a]	-	-
Beef, high moisture[a]	-	-
Beef, w/sorbitol[a]	-	-
Beef, w/mono- diglycerides[a]	-	-
Dates, low moisture	4.8	4.0-5.5
Dates, high moisture	0.0	-
Dates, w/acetyl glyceride coating	0.0	-
Peas, low moisture	4.9	3-8
Peas, high moisture	0.0	-
Cereal, low moisture[a]	-	-
Cereal, high moisture[a]	-	-
Nonfat milk solids	12.0	9-14.5
Potato chips[a]	-	-

a/ Could not drill holes for mounting bar without breaking apart.

8. Dispersibility in Water

a. Method: Dispersibility of the bars in water was tested by placing the bars in 100 ml. of water in a wide-mouth, 8-oz. bottle, and shaking with a lateral motion at 140 ± 10 strokes/min until complete dispersion had been effected. The volume of water used was approximately 10 times the volume of solids, except for the date bars, in which case it was about seven times. More rapid shaking than about 140 strokes/min resulted in considerable foaming, particularly with the nonfat milk solids bars, and shaking slower than 140 strokes/min gave little agitation. The test was conducted at room temperature.

b. Results: Table VII is a tabulation of dispersibility times for the various test bars. The beef bars all dispersed readily. The low moisture pea bars required 23 sec. to disperse compared with 6 sec. for the high moisture pea bars. Even though they were rather crumbly and had low hardness, shear and cohesiveness values, the cereal bars required about 3 hr. or more for dispersion. The milk and date bars underwent more of a dissolving than dispersing type action. It is possible that almost complete saturation of the solutions prolonged the dispersion times for the date and milk bars. A small core of material from the potato chip bars never reached complete dispersion, even after 8 hr.

9. Water Activity at 25°C

a. Method: Water activity of the food bars was determined with an Aminco-Dunmore electric hygrometer.* The leads for the sensing element receptacle of the instrument were sealed into a No. 6-1/2 rubber stopper. The material of which the water activity was to be measured was placed in a clean, dry 250-ml. Erlenmeyer flask. The proper sensing element was attached to the receptacle and suspended above the test material; the rubber stopper holding the receptacle leads served to seal the flask. The flask was then immersed in a water bath maintained at 25°C. Readings were taken at 15-min. intervals until the system had attained equilibrium as indicated by constant readings. The reading was converted to relative humidity by means of the calibration curves supplied for each sensing device, and the relative humidity was converted to water activity by dividing by 100. Generally, equilibrium was attained in

* American Instrument Company, Silver Spring, Maryland.

TABLE VII

MEASUREMENTS OF TIME IN MINUTES REQUIRED FOR THE DISPERSION OF COMPRESSED FOOD BARS IN WATER

(minutes for complete dispersion[a/])

Food Bar	Time in Minutes	
	Avg. Value	Range
Beef, low moisture	< 1	-
Beef, high moisture	< 1	-
Beef, w/sorbitol	< 1	-
Beef, w/mono-,diglycerides	< 1	-
Dates, low moisture	420	375-450
Dates, high moisture	273	270-300
Dates, w/acetyl glyceride coating	288	270-300
Peas, low moisture	23	22-25
Peas, high moisture	6	5-7
Cereal, low moisture	316	300-340
Cereal, high moisture	175	170-180
Nonfat milk solids	150	-
Potato chips	> 480	-

a/ Bar placed in approximately 10 volumes of water and shaken with lateral motion at 140 ± 10 strokes/min.

2 hr. or less; the lower the water activity, the longer the time needed to reach equilibrium.

Waldham and Halvorson[6] used this type method to measure the equilibrium vapor pressure of bacterial endospores. We measured the activity of several saturated salt solutions and obtained values which varied within ±3 per cent of the values given by Robinson and Stokes.[7]

b. Results: Table VIII is a summary of the water activity measurements obtained on the compressed food bars. The beef bars of low moisture content, 2 per cent, had a water activity of less than 0.1 while the high moisture beef bars, about 6 per cent moisture, had a water activity of 0.26. The value for the date bars was 0.4 when the moisture content was about 10 per cent and 0.5 to 0.6 when the moisture level was about 16 per cent. A moisture content of about 7 per cent in the pea bars gave an activity value of 0.4 compared to less than 0.1 if the moisture level was as low as 2 per cent. The low moisture dates and the high moisture cereal bars contained the same percentage of moisture but the material of each was such that the water activity of the high moisture cereal bar was considerably greater (0.6) than that of the low moisture date bars (0.4). The low moisture cereal bars contained only 4 per cent moisture and the water activity was lowered to 0.2. The non-fat milk solids bars had a low water activity, 0.1, and the potato chips had a relatively high value of 0.4.

A check of the water activity of the test bars was made in conjunction with the determination of equilibrium relative humidity curves. If a bar is placed in an atmosphere of a relative humidity which, when divided by 100, equals the activity coefficient of the water in the food bar, the bar will neither gain nor lose weight. However, the bar will give off or take up moisture if the value is less than or greater than the water activity of the food bar. If, then, samples of the compressed food bars are equilibrated in atmospheres of several different relative humidities for a few hours and the change in weight plotted versus the different relative humidities, it is possible to obtain a good estimate of the water activity of the bars. Figure 3 shows such plots for several of the food bars. The values thus obtained are in good agreement with those shown in Table VIII. This method is similar to the isopiestric method of Robinson and Sinclair,[8] but the equipment was not as elaborate. Further discussion of the procedure and equipment will be given in the discussion of the equilibrium relative humidity curves.

TABLE VIII

WATER ACTIVITY MEASUREMENTS OF COMPRESSED FOOD BARS

Food Bar	Activity at 25°C Avg. Value	Range
Beef, low moisture	0.05	-
Beef, high moisture	0.26	0.24-0.29
Beef, w/sorbitol	0.06	0.04-0.07
Beef, w/mono-, diglycerides	0.06	0.05-0.08
Dates, low moisture	0.40	0.39-0.41
Dates, high moisture	0.53	0.51-0.54
Dates, w/acetyl glyceride coating	0.57	0.53-0.60
Peas, low moisture	0.08	0.07-0.08
Peas, high moisture	0.36	0.33-0.38
Cereal, low moisture	0.18	0.16-0.21
Cereal, high moisture	0.62	0.60-0.64
Nonfat milk solids	0.12	0.11-0.19
Potato chips	0.40	0.38-0.43

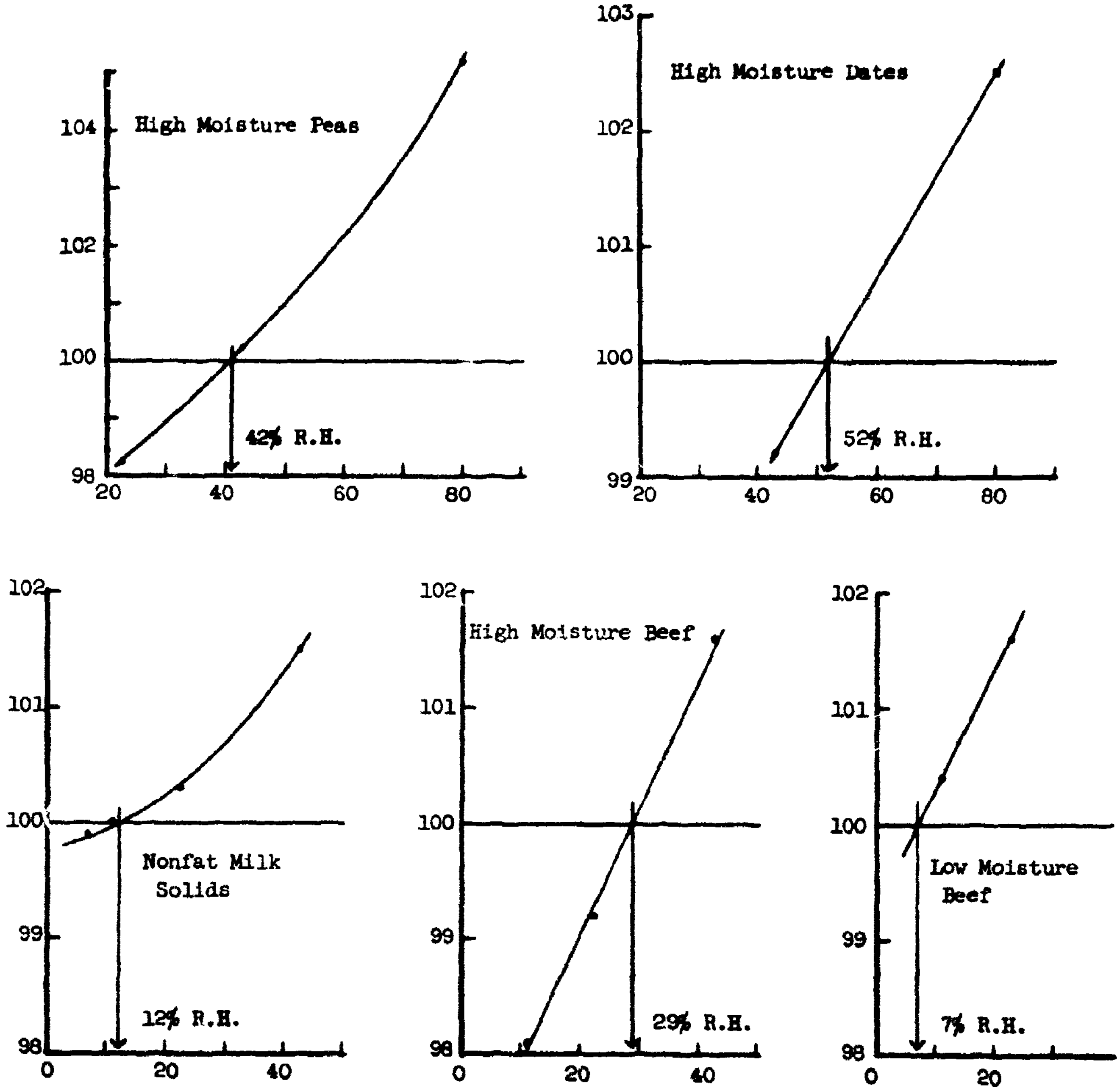

Fig. 3 - Estimation of Water Activity of Compressed Food Bars by Change in Weight after Exposure to Atmospheres of Controlled Relative Humidity. Vertical Axis, Sample Weight as Per Cent of Starting Weight; Horizontal Axis, Per Cent Relative Humidity of Atmosphere; Exposure Time, 66 Hr.; Water Activity = R.H./100.

The term "water activity" as herein used is the same as "equilibrium relative humidity" as used by Landrock and Proctor[9] or "moisture-vapor pressure" as used by Galwin.[10]

10. Elasticity

a. Method: Szczesniak[5] defines elasticity as "the rate at which a deformed material goes back to its undeformed condition after the deforming force is removed." We attempted to deform the food bars by compression between plates but found that most of the bars broke before any measurable deformation had been accomplished. Therefore, the determination of elasticity seems of little value as a compressed food bar characteristic.

b. Results: The high moisture date bars would undergo deformation. When subjected to a deformation of 5 per cent of their length, the recovery was 50 per cent of the deformation after 1 hr. The bars were held in the deformed state only as long as needed to measure, then released. If they had been held very long in the deformed state, they probably would have assumed it as a permanent configuration.

11. Permeability to Gas

a. Method: A simple apparatus was designed and the permeability to air determined after the ASTM procedure[11] for measuring the resistance of nonporous paper to passage of air. A diagram of the apparatus is shown in Fig. 4. A 100-ml. burette was suspended beneath the apparatus by an air-tight connection. A leveling bulb containing water was attached by means of a rubber hose. To start the test, a food bar was placed in the cavity of the apparatus and the top plate screwed down tightly. A soft rubber gasket was used to provide a seal at the outside edge of the apparatus and around the edge of the air hole in the top plate. The water level was brought to the zero point on the burette by raising the leveling bulb, and held at that point by clamping the rubber hose between the leveling bulb and burette. The leveling bulb was then placed in a lower support at a height that would allow the falling column of water to come to rest at the 75-ml. mark. The clamp was then released and the time measured for the water to fall to the 50-ml. mark. If the food bar to be tested was greater than 1/2 in. thick, a second

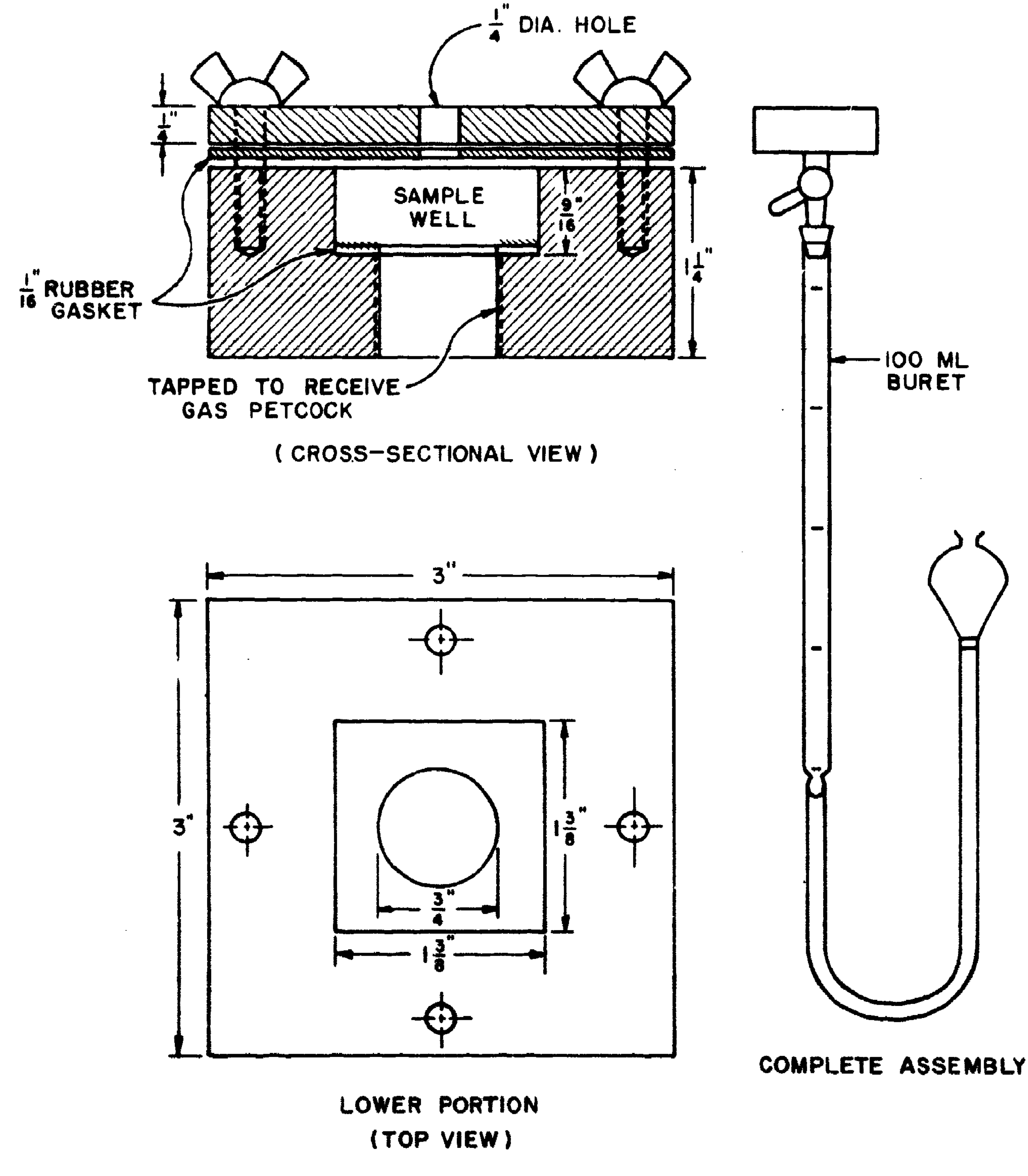

Fig. 4 - Diagram of Permeability Apparatus

rubber gasket, cut so as to slip over the projecting food bar, was placed on the apparatus to allow the top plate to be screwed down without crushing the food bar.

Since all burettes are not of the same diameter, the height of the water column before its release to pull air through the bar would be better standardized by specifying a distance between the zero point and the final resting point. This distance in our apparatus was 20 in.

b. Results: In Table IX are summarized the permeability times measured by the above procedure. The beef, cereal and potato chip bars were highly permeable. About 11 sec. were required for 50 ml. of air to be drawn through the nonfat milk solids bars and about 14 sec. for the pea bars. There was no measurable fall in the water column after 5 min. with the date bars in the apparatus and the term nonpermeable was applied to the date bars.

B. Chemical Characteristics

1. Moisture Content

a. Method: Moisture content was determined by drying in a vacuum oven. The loss in weight after 16 hr. at 65°C and about 50 mm. Hg pressure was taken as moisture loss. This time is longer than needed for some materials such as the beef, cereal, pea and nonfat milk solids bars, but there was no difference between weight loss at 8 - 10 hr. and 16 hr. The 16-hr. period is conveniently accomplished overnight. The dates were checked with an additional drying period of about 8 hr., making a total of 24, and the 24-hr. moisture content was about 1 per cent greater than the 16-hr. value, but the change with drying periods longer than 24 hr. was negligible. Thus, the exact time required for drying in the vacuum oven method will vary; 16 hr. are sufficient for most food materials.

b. Results: The moisture contents determined for the different food bars are shown in Table X. The moisture levels shown fall within the ranges specified in the contract except for the high-moisture beef bars, which were slightly higher than desired. The narrow range of values in each case indicates good duplication with replicate samples.

TABLE IX

PERMEABILITY TO AIR MEASUREMENTS ON COMPRESSED FOOD BARS

(time required to draw 50 cc. of air through bar)

Food Bar	Time, Sec.	
	Avg. Value	Range
Beef, low moisture	2.5	2.0-3.0
Beef, high moisture	2.3	2.0-3.0
Beef, w/sorbitol	2.4	2.0-3.0
Beef, w/mono- diglycerides	2.5	2.0-3.0
Dates, low moisture	Nonpermeable	
Dates, high moisture	Nonpermeable	
Dates, w/acetyl glyceride coating	Nonpermeable	
Peas, low moisture	13.8	11.0-19.5
Peas, high moisture	14.7	10.0-20.5
Cereal, low moisture	3.6	3.5-3.8
Cereal, high moisture	3.9	3.0-4.5
Nonfat milk solids	671	618-684
Potato chips	2.4	2.2-2.5

TABLE X

MOISTURE CONTENT OF COMPRESSED FOOD BARS

(loss in weight after overnight drying at 70°C and < 100 mm. Hg pressure)

Food Bar	Per Cent Avg. Value	Per Cent Range
Beef, low moisture	2.1	2.0-2.2
	2.5*	-
Beef, high moisture	6.4	6.2-6.9
	7.6*	-
Beef, w/sorbitol	1.6	1.4-1.9
	1.9*	-
Beef, w/mono- diglycerides	1.6	1.4-1.9
	1.9*	-
Dates, low moisture	9.6	9.4-9.8
Dates, high moisture	15.4	15.2-15.6
Dates, w/acetyl glyceride coating	16.4	15.7-16.8
Peas, low moisture	2.4	2.3-2.4
Peas, high moisture	6.8	6.7-7.0
Cereal, low moisture	3.9	3.7-4.2
Cereal, high moisture	9.5	9.3-9.6
Nonfat milk solids	3.5	3.4-3.5
Potato chips	5.6	5.5-6.3

* Fat-free basis, crude fat determined by petroleum ether extraction.

Several comparisons were made with the toluene distillation method and results agreed with those obtained by vacuum drying except for the date bars. Moisture by toluene distillation gave values for the date bars about 1 per cent greater than did the vacuum-oven method. Six hours were required for the dates in contrast to 1/2 to 1 hr. for any of the other bars for the toluene distillation method.

Thus, we recommend either the vacuum oven or toluene distillation methods for moisture determination.

We do not recommend the hot-air oven be used for drying food bar samples for moisture determination unless time and temperature conditions are specified which would give results which check with those determined by either vacuum oven or toluene distillation. In our experience, drying periods of up to several days at 100°C did not bring samples to constant weight, and obvious deterioration and loss of volatiles occurred if the temperature was 105°C or greater.

2. Free and Bound Lipid Content

a. Method: Free or crude fat was determined by solvent extraction in a Soxhlet apparatus as outlined by AOAC standard methods.[12] Comparable results were obtained with either ethyl ether or petroleum ether;* therefore, petroleum ether was used instead of ethyl ether because it is less hazardous. All samples were dried and ground before extraction. Free fat values are summarized in Table XI.

Bound lipid was calculated as the difference between total and free lipid, but the method used for determining total lipid was found to be in error for some of the food bars. We used chloroform:methanol, 2:1 volume ratio, extraction in a Soxhlet extractor to determine total lipid.[13] The method gave no apparent discrepancies with the beef, cereal, potato chip and nonfat milk solids bars; but values obtained for the date and pea bars were considerably greater than those reported by Watt and Merrill.[14] It was obvious that more than just lipid was being extracted by the chloroform:methanol solution. Therefore, the bound lipid values shown in Table XI are labeled apparent bound lipid.

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* Skellysolve-B

TABLE XI

FREE LIPID AND APPARENT TOTAL AND BOUND LIPID CONTENT OF COMPRESSED FOOD BARS

(per cent of dry weight)

Food Bar	Free Lipid*		Apparent Total Lipid**		Bound Lipid***
	Avg.	Range	Avg.	Range	
Beef, low moisture	16.2	15.6-17.3	20.6	18.8-22.0	4.4
Beef, high moisture	16.2	15.4-17.0	20.5	19.0-21.7	4.3
Beef, w/sorbitol	16.8	15.5-17.8	24.5	23.6-26.0	7.7
Beef, w/mono-diglycerides	17.6	16.9-18.3	25.2	22.5-27.5	7.6
Dates, low moisture	0.7	0.5-0.9	-	-	-
Dates, high moisture	0.6	0.5-0.6	39.4	25.5-48.3	38.8
Dates, w/acetyl glyceride coating	1.9	1.6-2.3	21.0‡	19.7-22.0	19.1
Peas, low moisture	2.9	2.7-3.1	17.9	16.8-20.1	15.0
Peas, high moisture	3.2	2.9-3.3	14.6	11.2-17.2	11.4
Cereal, low moisture	14.5	14.4-14.7	17.5	17.1-18.0	3.0
Cereal, high moisture	13.9	13.3-14.3	17.1	15.8-17.4	3.2
Nonfat milk solids	1.4	0.3-2.0	2.1	1.4-2.6	0.7
Potato chips	31.8	31.4-32.0	32.3	31.8-32.6	0.5

* Determined by petroleum ether extraction.
** Determined by chloroform:methanol extraction, 2:1 volume ratio.
*** Difference between free and total lipid.
‡ Sample was not dried prior to extraction.

b. Results: Beef bars with added mono- and diglycerides had higher free lipid values than the other three types. This result was expected, and the lipid values should have been 2 per cent greater than those of the other three. The apparent bound lipid for the low and high moisture beef was about 4.4 per cent, while the group with sorbitol added had a value of 7.7 per cent. The free fat values for the dates and peas were in agreement with the values given by Watt and Merrill.[14] The total fat, as determined by the chloroform:methanol extraction, and consequently the bound fat values, were considerably divergent. These results indicated the methanol component of the solvent mixture was probably extracting some of the sugar from the date bars and sorbitol from the pea bars. The pea bars consisted of 10 per cent sorbitol. This supposition is further supported by the fact that the bound lipid of the sorbitol beef bars was about 3 per cent greater than the beef bars containing no sorbitol.

The free fat of the cereal bars was found to be about equal to the percentage of shortening added and the values for the potato chip and nonfat milk solids bars were as expected. The bound lipid for the potato chip and nonfat milk solids was less than 1 per cent and the cereal bars about 3 per cent.

Two approaches were tried in an attempt to circumvent the problem of the chloroform:methanol extracting nonlipid materials. We first attempted a pre-extraction with water to remove a large portion of the water-soluble material. This did not prove satisfactory. A second approach was to extract with the chloroform:methanol in the regular fashion, then extract the chloroform:methanol with water. In so doing, we added magnesium chloride to retard emulsion formation as used by Winter[15] in a similar procedure. The solvent mixture was then transferred to a dry, tared flask and the solvent evaporated to determine the amount of fat extracted. This procedure was not tested extensively, but the following percentages for total fat content were obtained: low moisture beef, 18.0; dates with acetyl glyceride coating, 2.5 and 3.8; low moisture dates 0.4 and 0.3; beef with mono- and diglycerides, 20.1, 20.1, 19.8; and high-moisture peas, 4.6, 4.6, and 4.8 (each value represents a different sample). These values were not checked by any other procedure, but the date and pea values are more reasonable than the results obtained by the simple extraction method.

The procedure in greater detail was as follows. From 5 - 10 g. of sample was weighed into a paper extraction thimble (the samples were used as is, without additional drying but crushed or cut into small pieces). They were extracted for 4 hr. with chloroform·methanol solution (2:1 volume ratio) in a Soxhlet extractor. Approximately 150 ml. of solvent was used. After cooling to room temperature, the solvent was quantitatively transferred to a 500-ml. separatory funnel. Approximately 50 ml. of water was then added with 1 ml. of molar magnesium chloride solution and the mixture shaken, allowed to stand until the chloroform: methanol separated. The organic solvent was then drained into a clean, dry, tared 250-ml. flask (several dry boiling chips were tared with the flask). The water solution was extracted twice more with 50-ml. portions of fresh chloroform:methanol solution. The chloroform:methanol was then evaporated under gentle heat and vacuum. Final evaporation and drying was accomplished in the vacuum oven, after which the flask and fat residue were allowed to cool in a desiccator and weighed. We think that this method will yield a more authentic value for total lipid content, and warrants further investigation for reliability and accuracy. It is similar in many respects to the procedure reported by Winter. 15/

3. Reducing Sugars

a. Method: The reducing sugar content was determined after the method of Moyer and Holgate. 16/ The major departure from the method as outlined by these authors was the extraction procedures. The extraction procedure recommended by the AOAC 17/ was used instead. Reagents were made as specified in the appropriate references.

Five to ten grams of material were placed in a clean 250-ml. volumetric flask and 125 ml. of 50 per cent ethanol were added. The flask and contents were placed in an 85°C water bath for 1 hour, then allowed to stand overnight and made to volume with 95 per cent ethanol, and mixed. A 5- or 10-ml. aliquot of the alcoholic extract was transferred to a clean test tube and evaporated to near dryness in a boiling water bath. The walls of the test tube were washed down with 2 - 3 ml. of water and 2 ml. of barium hydroxide solution added and mixed well, followed by the dropwise addition of 2 ml. of zinc hydroxide solution. The contents of the tube were quantitatively transferred to a filter and the filtrate collected in a 50- or 100-ml. volumetric flask. An aliquot of the clarified extract estimated to contain 0.5 - 1.5 mg. of glucose, or its equivalent in reducing power, were pipetted into a clean Folin-Wu sugar tube with 2 ml. of reagent, mixed well and placed in a boiling water bath for 20 minutes. After cooling, 2 ml. of arsenomolybdate solution was added, water added to the 25-ml. mark and the contents of each tube mixed by inversion. After 15 min., the absorbancy of the solutions was read in a colorimeter* at a wavelength of 530 mμ. The absorbancy was converted to glucose concentration by comparison with a standard glucose solution run as prescribed at the same time. Appropriate dilution factors were applied and the reducing sugar content calculated as the per cent glucose equivalent. The sample size, dilutions and aliquot for color development vary according to the estimated reducing sugar content of the material. For example, in the case of dates, 5 g. of dry matter were extracted, 5 ml. were clarified and the filtrate made to 100 ml.; a 5-ml. aliquot of this dilution was transferred to a 100-ml. flask made to volume and 2 ml. of this final dilution used for the color development; potato chips, 10 g. of dry matter extracted, 10 ml. clarified and filtrate made to 50 ml., 2 ml. of which were used for color development.

b. Results: Table XII is a summary of the results obtained. The beef, pea, and potato chip bars were quite low in reducing sugars while about 75 per cent of the dry matter of the dates consisted of reducing sugar. The value shown for the nonfat milk solids, when doubled, is equivalent to the percentage of lactose expected. The nonfat milk solids contributes the reducing sugar found in t. cereal bars.

The relatively narrow range of values shown for the date, cereal and nonfat milk solids bars indicates good duplicability. Percentagewise, the range was greater when the reducing-sugar content was low as in the beef, pea, and potato chip bars. Recovery of glucose added to date samples was good. Recovery of added glucose at the low levels determined for beef and peas was not as good. With the aliquots used, the absorbancy of the colored solutions from the beef, pea, and potato chip bars was only about 0.15 unit greater than the blank. We did not investigate extensively, but evaporation of larger aliquots of the alcoholic extract to give a higher absorbance value in the colored solution showed promise of improving the accuracy of the determination in these instances.

* Bausch and Lomb, Spectronic-20.

TABLE XII

REDUCING SUGAR CONTENT OF COMPRESSED FOOD BARS

(per cent glucose equivalent on dry matter basis)

Food Bar	% of Dry Matter Avg. Value	% of Dry Matter Range
Beef, low moisture	0.24	0.11-0.32
Beef, high moisture	0.38	0.16-0.44
Beef, w/sorbitol	0.15	0.06-0.22
Beef, w/mono- diglycerides	0.20	0.11-0.26
Dates, low moisture	73.8	73.5-74.1
Dates, high moisture	77.5	76.0-78.4
Dates, w/acetyl glyceride coating	78.0	75.8-80.1
Peas, low moisture	0.2	-
Peas, high moisture	0.38	0.37-0.40
Cereal, low moisture	6.4	6.3-6.6
Cereal, high moisture	6.6	-
Nonfat milk solids	26.3	25.0-28.4
Potato chips	0.2	0.1-0.2

4. pH

a. Method: The electrometric method recommended by the AOAC[18] was used to determine the pH of a water suspension of the food bars. The procedure was used as outlined by the AOAC except that the instrument* was standardized with a pH 7.0 commercially prepared buffer.**

b. Results: The pH values obtained are summarized in Table XIII. No range is given because the values did not vary more than ±0.1 pH unit, and in most cases all samples of a given group showed no variation. The beef, date, and potato chip bars had values of less than 6.0; the value of 5.6 for the dates coated with acetyl glycerides was the lowest. The cereal and nonfat milk solids bars had a pH of about 6.5, while the pea bars were 7.0.

5. Water-Soluble Fraction

a. Method: Water-soluble fraction was determined by the method given by Jacobs[19] for preparing cold-water extracts of meat and meat products. The only alteration was in the water temperature; instead of water at 40°F, water at room temperature, 78 ± 2°F, was used.

An aliquot of the food bar was accurately weighed and placed in a clean 50-ml. centrifuge tube. About 40 ml. of distilled water was added and the mixture allowed to stand with occasional stirring for 10 min. The tube was centrifuged and the supernatant filtered through Whatman No. 31 filter paper. This was repeated twice with 50 ml. of water and four times with 25 ml. of water, the residue being washed into the filter paper with the last 25 ml. The residue and filter paper were washed three times. The filtered extract was diluted to a volume of 500 ml. in a volumetric flask and mixed well. An aliquot of the extract, 100 or 200 ml., was transferred to a tared evaporating dish and evaporated to dryness to determine solids extracted. Initial drying of the extract was accomplished in a 100°C oven and the final drying in the vacuum oven. From the total solids extracted the water-soluble fraction of the dry matter was calculated.

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* Leeds and Northrup Company, Philadelphia, Pennsylvania, Assembly No. 7663-A1.

** Beckman Buffer Solution 3581, Beckman Instruments Incorporated, Fullerton, California.

TABLE XIII

THE pH OF A WATER SUSPENSION OF COMPRESSED FOOD BARS

Food Bar	pH Avg. Value
Beef, low moisture	5.7
Beef, high moisture	5.7
Beef, w/sorbitol	5.7
Beef, w/mono-,diglycerides	5.7
Dates, low moisture	5.8
Dates, high moisture	5.8
Dates, w/acetyl glyceride coating	5.6
Peas, low moisture	7.0
Peas, high moisture	7.0
Cereal, low moisture	6.4
Cereal, high moisture	6.4
Nonfat milk solids	6.5
Potato chips	5.8

The supernatant liquid sometimes filtered slowly. The nonfat milk solids were particularly hard to filter. The No. 31 Whatman filter paper - which is coarse - aided filtration. Another manipulation which aided was to reduce the original sample size and evaporate a larger aliquot of the extract. Representative sample sizes and aliquots evaporated were as follows: beef bars, 3 g., 200 cc.; date bars, 5 g., 100 cc.; pea bars, 5 g., 200 cc.; cereal bars, 12 g., 100 cc.; nonfat milk solids, 3 g., 200 cc.; and potato chip bars, 6 g., 200 cc.

b. Results: Compression had virtually no effect on the solubility of the nonfat milk solids; essentially 100 per cent of the dry matter was water soluble (see Table XIV). Since they contained 0.5 per cent of magnesium stearate, we did not expect a value greater than 99.5 per cent. The potato chip bars had the lowest water-soluble fraction, 17 per cent followed in order by the beef bars, 25 - 30 per cent; cereal and pea bars, about 40 per cent; and the date bars 85 - 90 per cent. The range of values for replicate samples showed results could be duplicated.

6. Equilibrium Relative Humidity Curve and Rate of Equilibration to 40 Per Cent R.H.

a. Method: The equilibrium relative humidity curves for the food bars were established after the method of Funk.[20] Samples of the test bars were placed in a tared weighing bottle and placed in closed containers in which the relative humidity of the atmosphere was controlled by saturated salt solutions. The saturated salt solutions were chosen to be as close as possible to relative humidities of 5, 10, 20, 40, and 80 per cent. The different salt solutions used and the relative humidity of the atmosphere at 78°F (Robinson and Stokes[7]) over them were sodium hydroxide, 7.0 per cent; lithium chloride, 11.0 per cent; potassium acetate, 22.4 per cent; potassium carbonate, 42.8 per cent, and potassium bromide, 80.7 per cent. The containers were kept at room temperature, which was 78 ± 2°F. The bottle and sample were weighed periodically and the sample was considered at equilibrium if the weight change over a three-day period was 1 mg. or less per gram of sample. After samples had reached equilibrium, the equilibrium moisture content was determined by drying in the vacuum oven.

TABLE XIV

WATER-SOLUBLE FRACTION OF COMPRESSED FOOD BARS

(per cent of dry matter)

Food Bar	Per Cent Avg. Value	Per Cent Range
Beef, low moisture	27	[illegible]
Beef, high moisture	27	[illegible]
Beef, w/sorbitol	29	28-30
Beef, w/mono-, diglycerides	29	26-32
Dates, low moisture	86	84-88
Dates, high moisture	89	88-90
Dates, w/acetyl glyceride coating	86	86-87
Peas, low moisture	41	40-42
Peas, high moisture	42	40-45
Cereal, low moisture	41	40-41
Cereal, high moisture	38	38-39
Nonfat milk solids	99	97-100
Potato chips	17	16-18

b. Results: Figures 5-10 show the equilibrium relative humidity curves for the compressed food bars studied. Except for the date bars, the equilibrium moisture content was 5 per cent or less at relative humidities of up to 40 per cent. In no case was the value greater than 10 per cent at 42.8 per cent R.H. The equilibrium moisture content at 80 per cent R.H. was two to three times that at 40 per cent R.H. Difference in initial moisture level of a given bar type had little influence on the equilibrium moisture levels.

The rate of equilibration to 40 per cent R.H. was followed by checking the change in weight in an atmosphere of 42.8 per cent R.H., maintained over a saturated potassium carbonate solution. The weight was checked at close enough intervals, especially over the first week of equilibration, so that a plot could be made relating the food bar weight after successive periods of equilibration to the starting weight. Whole food bars were used since their rate of equilibration tended to be slower than that of pieces. The equilibrium moisture content of whole bars or pieces was the same.

Figures 11-15 show the rates with which the various compressed food bars equilibrated to 42.8 per cent R.H. The beef bars required 7 - 10 days to reach equilibrium. The low moisture dates had a water activity of about 0.4, and reached equilibrium in just a few days. The other date bars were much longer in attaining equilibrium. The high moisture pea bars required about one week, but the low moisture pea bars required 12 - 14 days. The cereal bars needed 12 - 14 days and the non-fat milk solids almost three weeks to attain equilibrium.

7. Rate and Amount of Gaseous Oxygen Uptake

a. Method: Oxygen uptake was determined by means of the Warburg respirometer. An accurately weighed sample, usually an intact bar or its weight equivalent, was placed in a 200-ml. reaction flask. The flask was flushed with oxygen for 1 min. and stoppered until it was attached to the manometer. The constant temperature bath was maintained at 50 ± 0.2°C. One hour was allowed for temperature equilibration before the manometer was closed to the outside atmosphere and the initial reading made. Each day the manometers were read and adjusted back to atmospheric pressure if the change in the level of manometer fluid in the open arm was more than 10 mm. During periods of rapid change in air pressure, it was sometimes necessary to read and adjust the manometers more often to keep the fluid levels on scale.

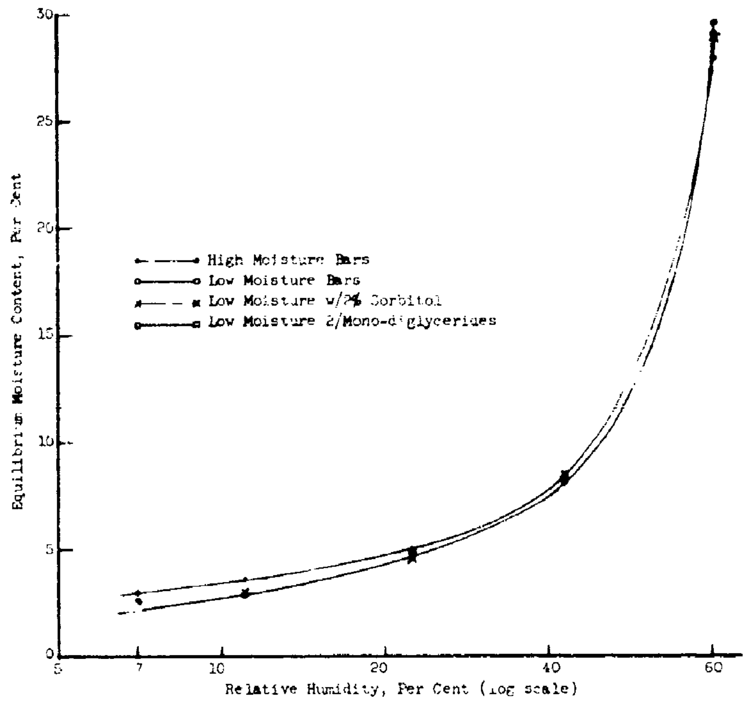

Fig. 5 - Equilibrium Relative Humidity Curves for Beef Bars

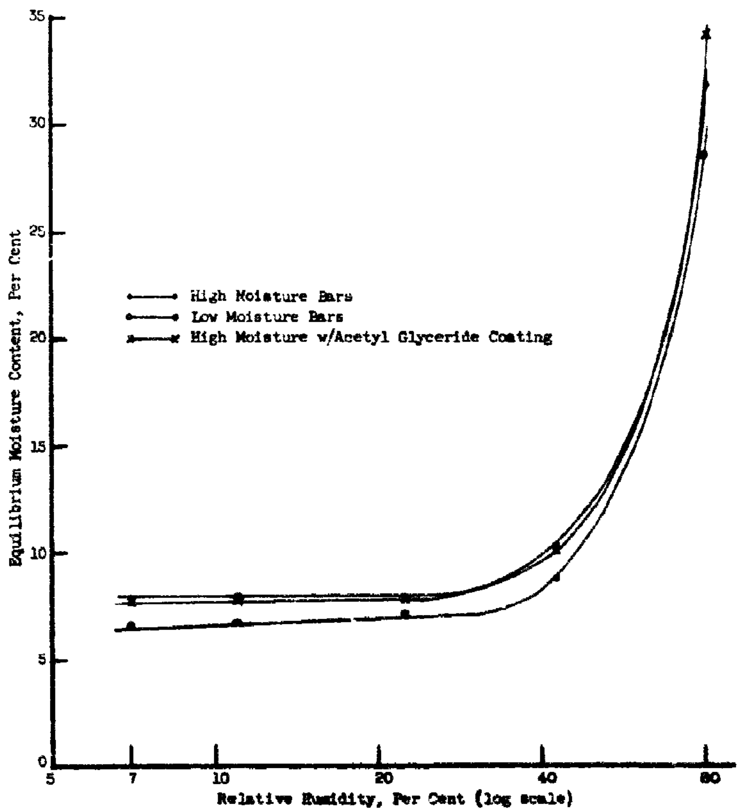

Fig. 6 - Equilibrium Relative Humidity Curves for Date Bars

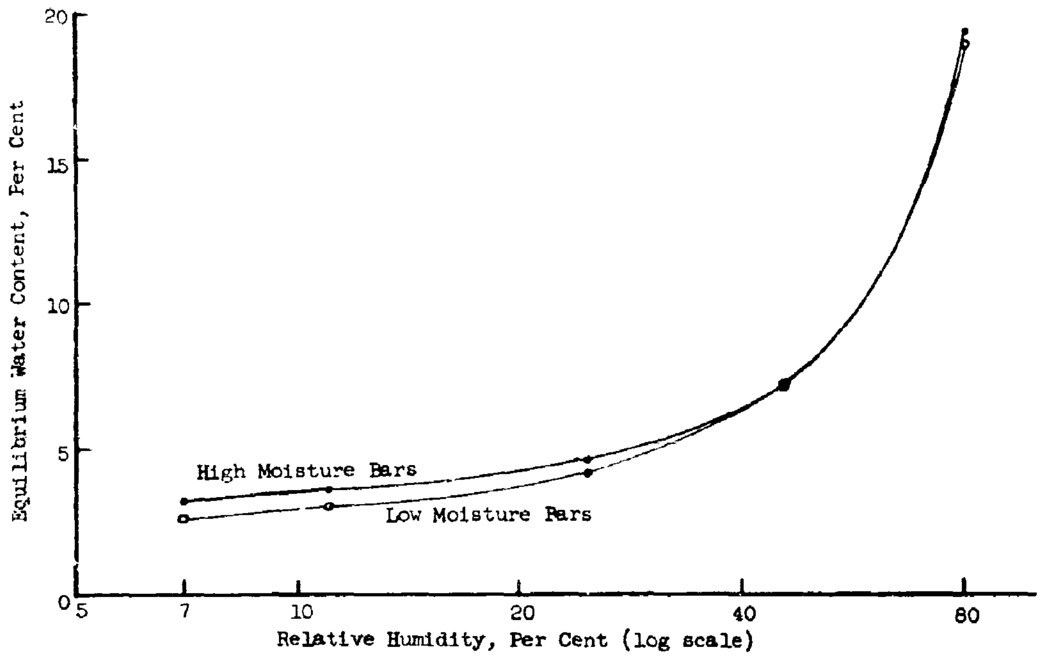

Fig. 7 - Equilibrium Relative Humidity Curves for Cooked, Freeze-Dried Pea Bars

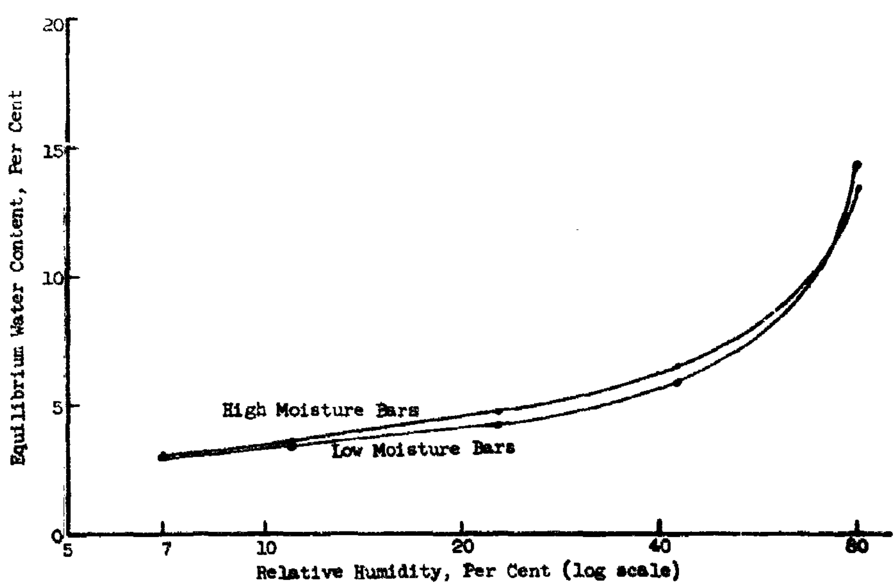

Fig. 8 - Equilibrium Relative Humidity Curves for Cereal Bars

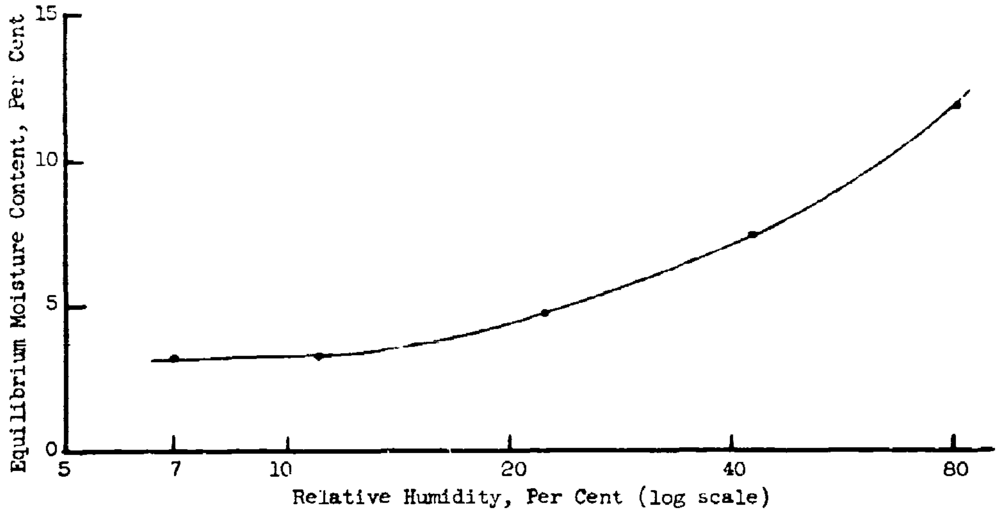

Fig. 9 - Equilibrium Relative Humidity Curve for Nonfat Milk Solids Bar

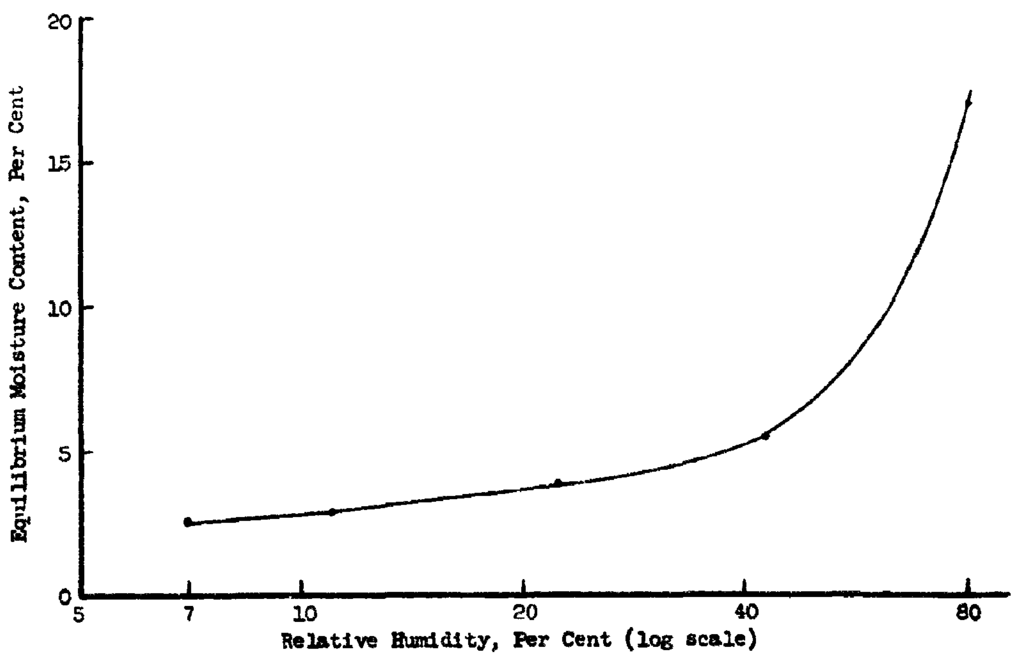

Fig. 10 - Equilibrium Relative Humidity Curve for Potato Chip Bars

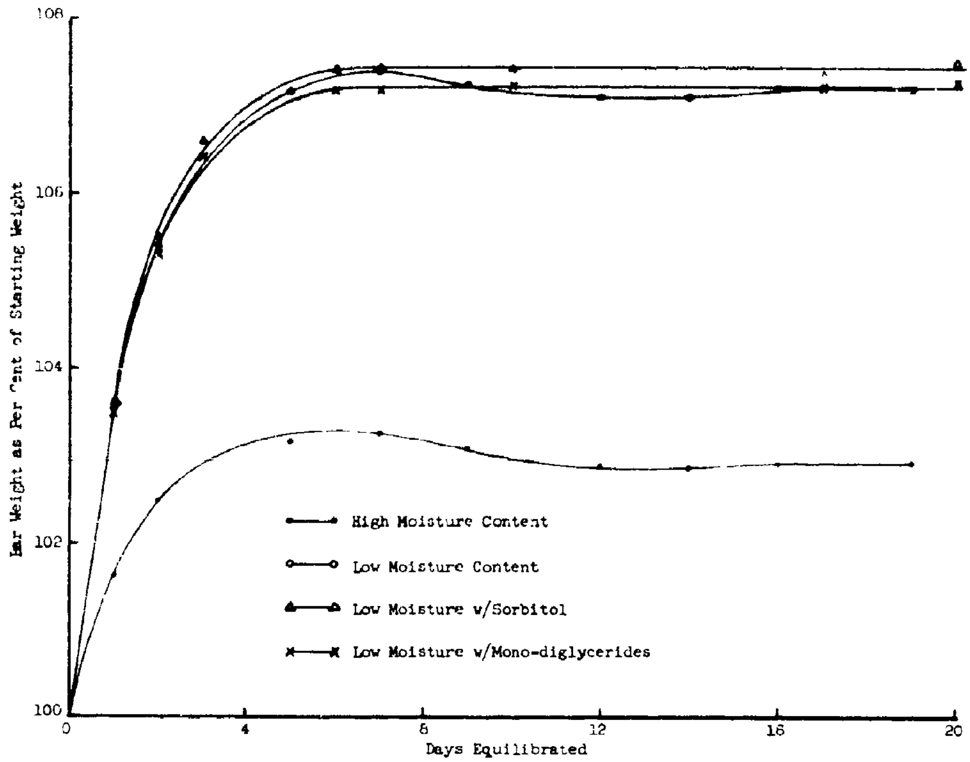

Fig. 11 - Rate of Equilibration of Beef Bars to 42.8 Per Cent Relative Humidity

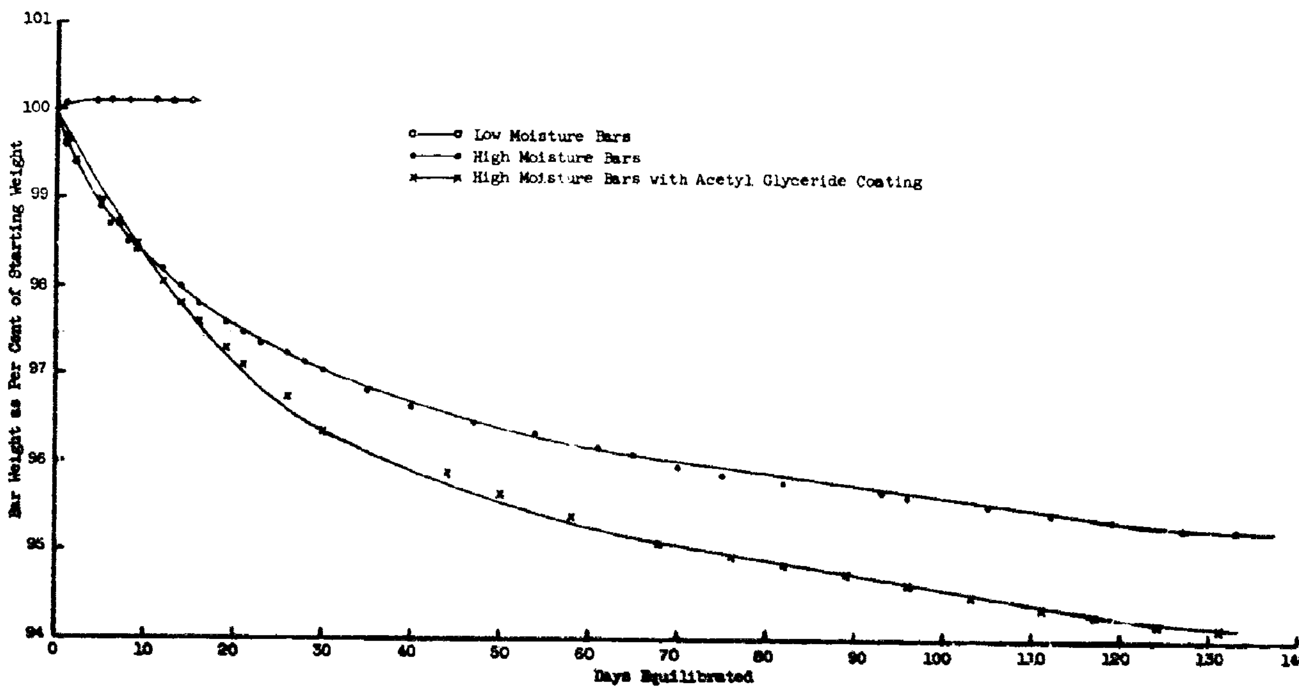

Fig. 12 - Rate of Equilibration of Date Bars to 42.8 Per Cent Relative Humidity

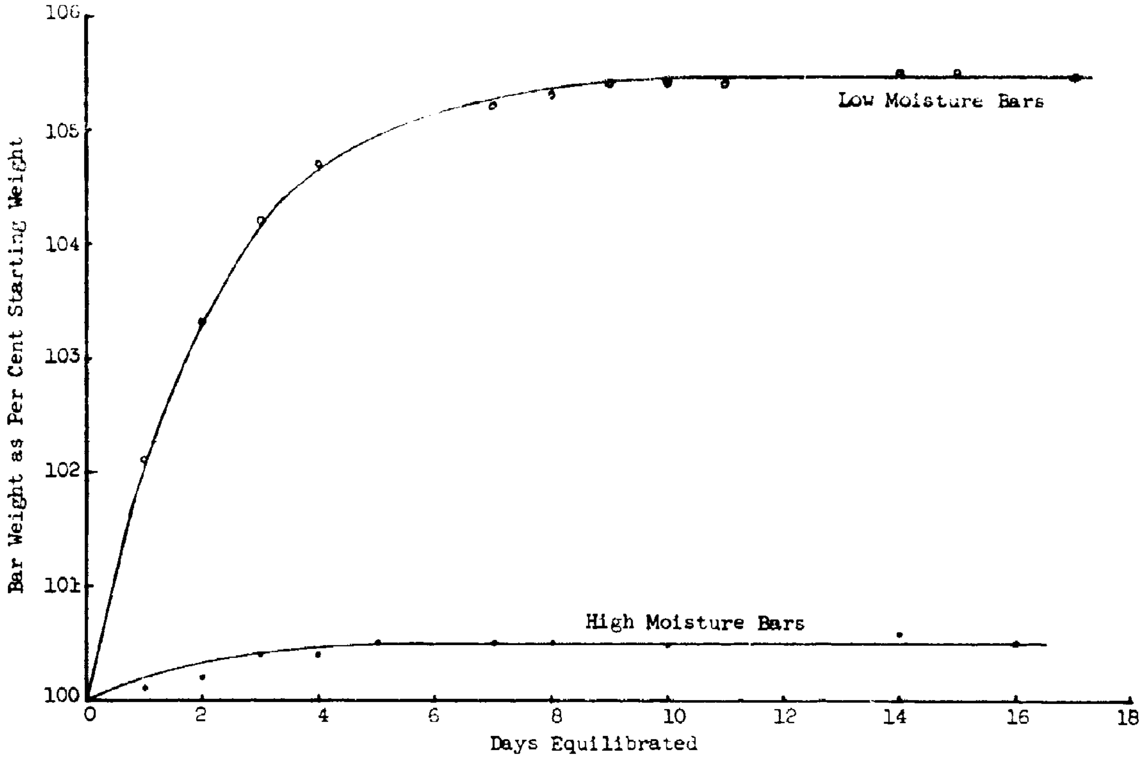

Fig. 13 - Rate of Equilibration of Pea Bars to 42.8 Per Cent Relative Humidity

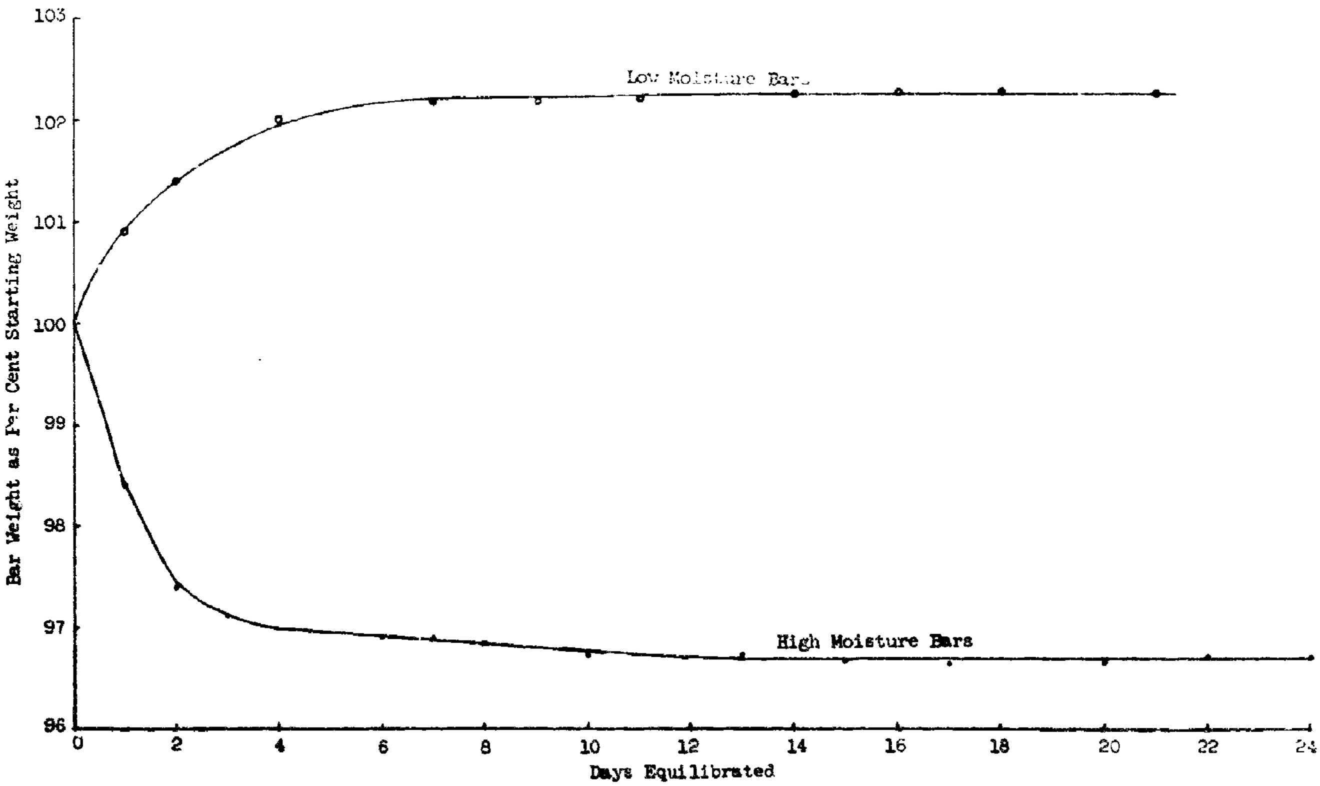

Fig. 14 - Rate of Equilibration of Cereal Bars to 42.8 Per Cent Relative Humidity

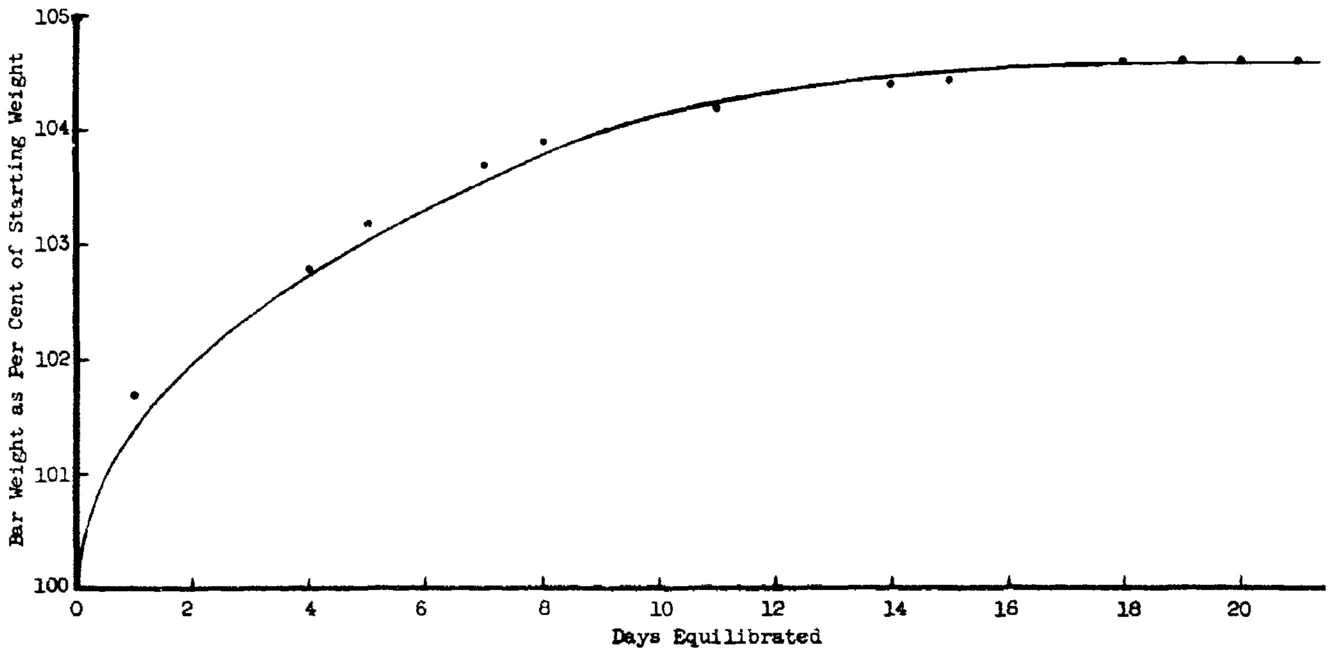

Fig. 15 - Rate of Equilibration of Nonfat Milk Solids Bars to 42.8 Per Cent Relative Humidity

The manometers were brought back to atmospheric pressure by opening the closed arm of the manometer to the atmosphere and readjusting the fluid level to the starting point. Thus, the oxygen concentration was diluted over the observation period. A 10-day observation period was carried out.

b. Results: Figures 16-20 show the accumulative uptake of oxygen for the freshly prepared food bars as determined by the above procedures. The high and low moisture beef bars took up more oxygen than did the bars with sorbitol and mono-,diglycerides added. The latter bars were similar to the low moisture bars with respect to treatment and in composition except that sorbitol and mono-,diglycerides were added. We did not attempt to elucidate the mechanisms causing the marked differences in oxygen uptake. The low and high moisture beef bars were run more than the regular 10-day observation period and as the rate of uptake was decreasing, the rates of uptake continued to decrease up to the 16th day at which time the trial was discontinued. However, as shown in Fig. 16, both types of bars were still taking up some oxygen. The low and high moisture dates took up oxygen at a much slower rate than did any of the beef bars, less than 200 µl/g in 10 days. However, the bars with acetyl glyceride coatings took up more than 700 µl. in the first 10 days observed. These, too, were run longer than the usual 10 days and their rate of uptake tended to decrease, but not cease, from the 10th to 16th day of observation. Approximately 450 µl. of oxygen per gram of dry matter were taken up by the pea bars, 250 µl. by the low moisture cereal bars, and 400 µl. by the high moisture cereal bars over the 10 days observed. The potato chip bars had taken up about 250 µl/g and the nonfat milk solids approximately 275 µl/g in 10 days of observation. The low moisture beef bars and the nonfat milk solids bars were the only ones which showed any definite decrease in the rate of uptake over the 10-day observation period.

In general, the high moisture group of each type of bar tended to have a greater rate of uptake than did the low moisture group. In no instance did any of the bars reach an apparent saturation with oxygen in the 10 days observed. The decrease in the rate of uptake by the two groups of beef bars as well as the dates with acetyl glyceride coatings was not caused by dilution of the oxygen in the reaction flasks with atmospheric air. Two flasks of each of these groups were removed from the bath after 10 days and flushed with oxygen, as they had been at the start of the trial. They continued to take up oxygen from the 10th to 16th days at the same rate as the ones which had not been reflushed with oxygen.

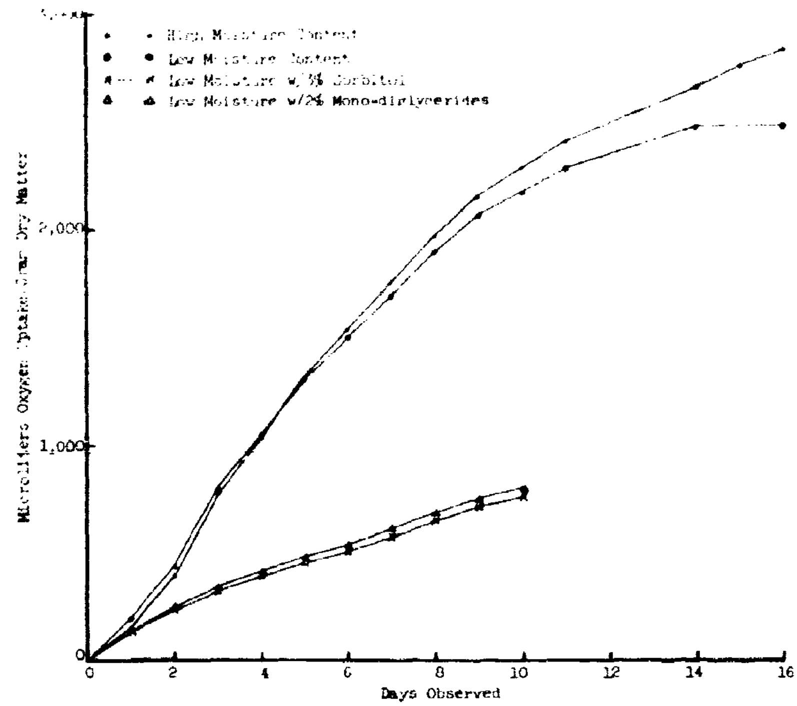

Fig. 16 - Oxygen Uptake by Beef Bars. Atmosphere of Approximately 100 Per Cent Oxygen at 50°C.

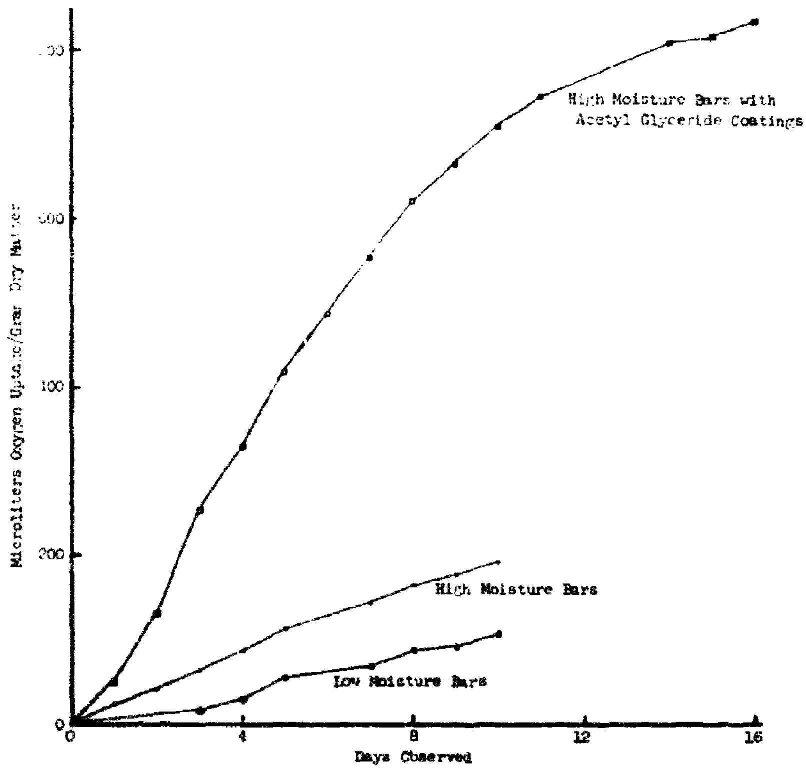

Fig. 17 - Oxygen Uptake by Date Bars. Atmosphere of Approximately 100 Per Cent Oxygen at 50°C.

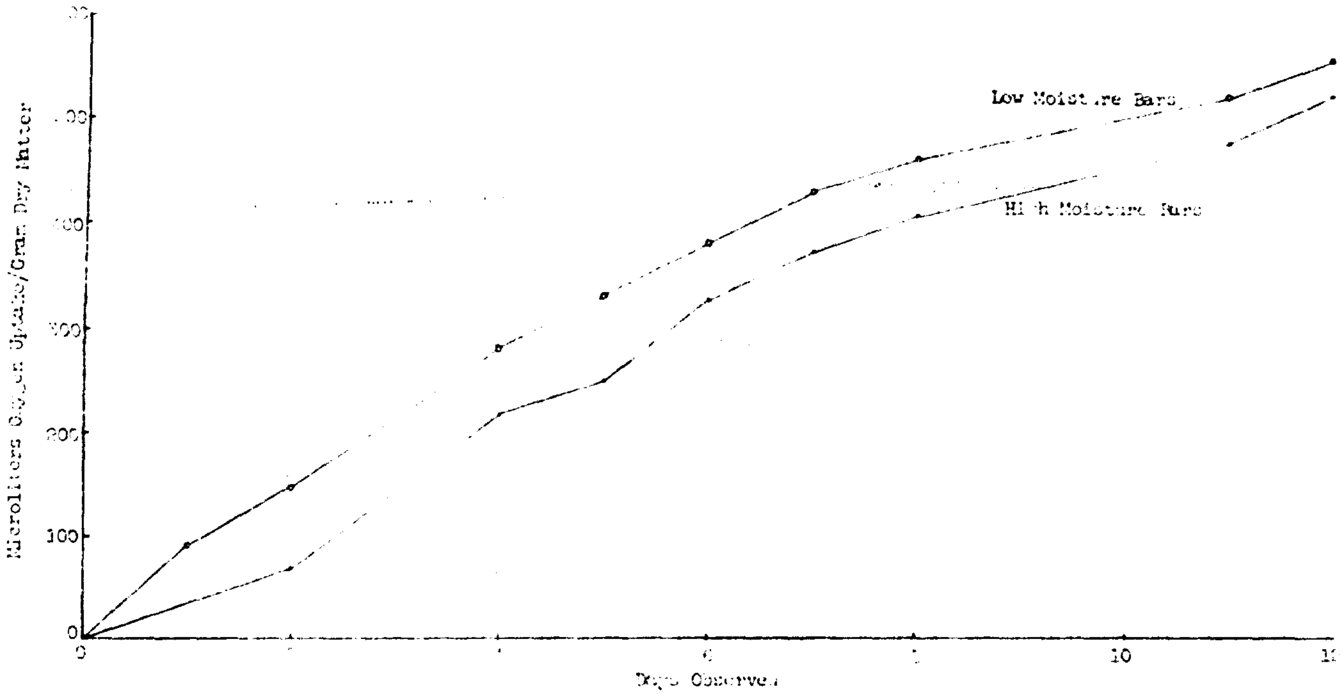

Fig. 18 - Oxygen Uptake by Pea Bars. Atmosphere of Approximately 100 Per Cent Oxygen at 50°C.

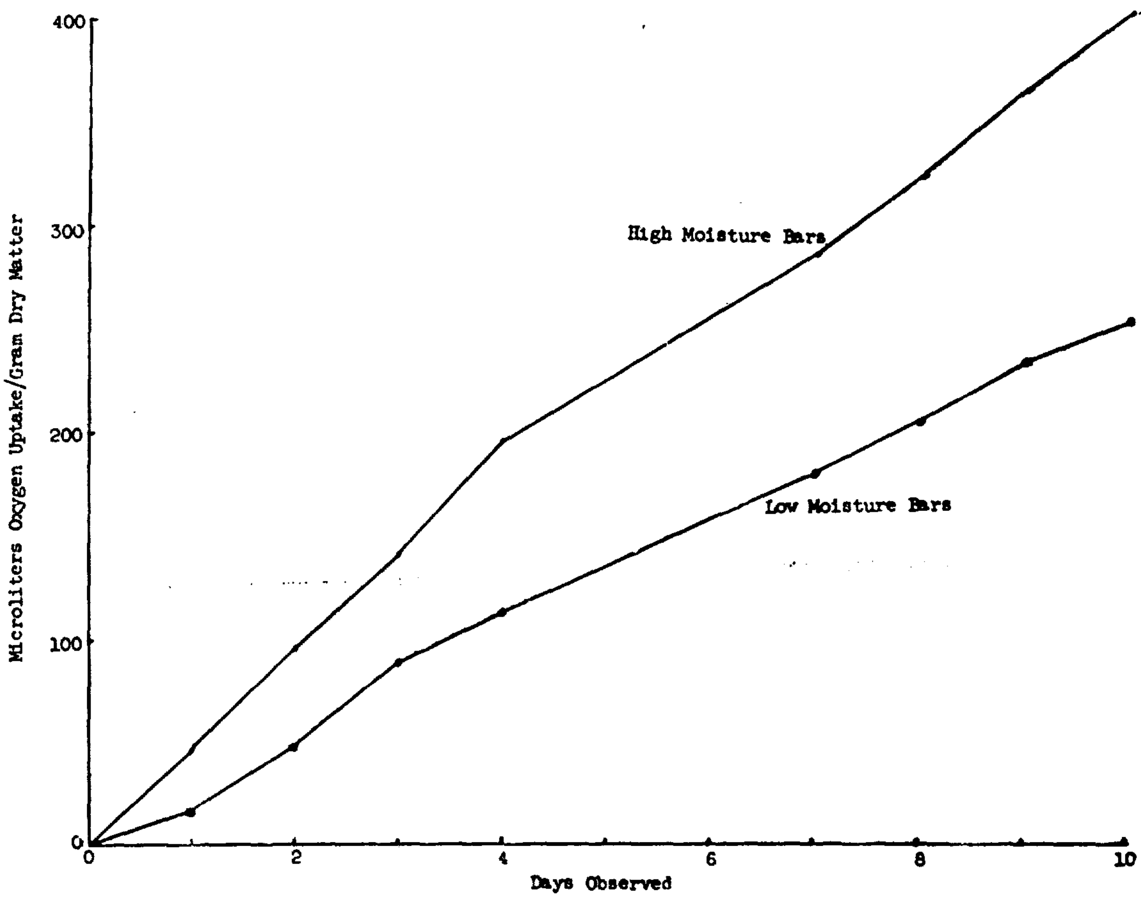

Fig. 19 - Oxygen Uptake by Cereal Bars. Atmosphere of Approximately 100 Per Cent Oxygen at 50°C.

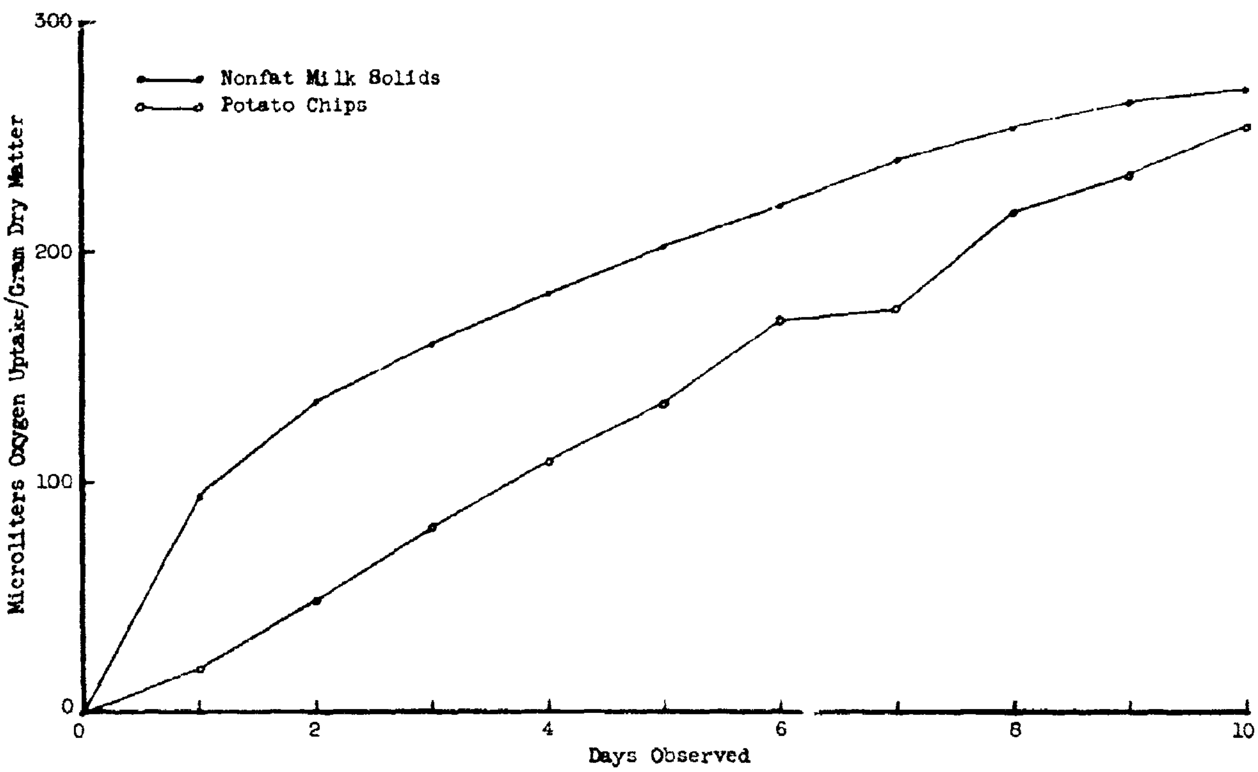

Fig. 20 - Oxygen Uptake by Nonfat Milk Solids and Potato Chip Bars. Atmosphere of Approximately 100 Per Cent Oxygen at 50°C.

At 50°C each microliter of oxygen is equivalent to approximately 0.038 μM of oxygen (assuming barometric pressure of 760 mm.). Thus, the 10-day uptakes recorded in these experiments ranged from about 76 μM/g for the beef bars to about 3.8 μM/g for the low moisture date bars.

8. Changes Associated with Oxygen Uptake; Peroxide Number, Carbonyl Content, Thiobarbituric Acid Number

Three different chemical characteristics; (1) peroxide number; (2) carbonyl content; and (3) thiobarbituric acid number, were used to reflect changes in the compressed food bars associated with oxygen uptake. The following procedures were used with minor changes, principally in sample extraction, for the respective determinations, peroxide number, procedure recommended by the Committee on Analysis of Commercial Fats and Oils;[21] carbonyl content, that reported by Henick, Benca and Mitchell;[22] thiobarbituric acid number, the procedure of Sidwell et al.[23] Reagents and solutions were prepared as directed in the respective references.

a. Method:

(1) Peroxide number: For the determination of peroxide number, approximately a 5-g. sample was accurately weighed into a glass-stoppered 50-ml. centrifuge tube. The sample was extracted with 30 ml. of acetic acid-chloroform solution, centrifuged, and the supernatant filtered through a coarse filter paper into a clean, dry 250-ml. Erlenmeyer flask. The extraction was repeated with two 20-ml. portions of acetic acid-chloroform solution. The acetic acid-chloroform extract was then treated with potassium iodide and water and the liberated iodine titrated with standard thiosulfate as directed by the Committee on Analysis of Commercial Fats and Oils.

(2) Carbonyl content: For the carbonyl determination, 3 - 5 g. of the ground food bars were accurately weighed into a 50-ml. glass-stoppered centrifuge tube and 25 ml. of carbonyl-free benzene were added. The tube was shaken, allowed to stand several minutes, shaken again and centrifuged. An aliquot of the supernatant estimated to contain up to 1 μM of total carbonyl was pipetted into a clean, dry 50-ml. volumetric flask and the color developed as directed.[22] The absorbancy

of the [illegible] in a colorimeter* and the concentration of saturated [illegible] was calculated by means of simultaneous equations. [illegible] for the simultaneous equations were derived from [illegible] standard solutions, 1 µM in the color development [illegible] dinitrophenylhydrazones of butyraldehyde and crotonaldehyde. [illegible] these two aldehydes were prepared and recrystallized [illegible] melting point range of 2°C. An accurately weighed aliquot of [illegible] recrystallized 2,4-D was dissolved in carbonyl-free benzene to make the standard solutions.

(5) [illegible] acid number: If the aliquot required for the carbonyl [illegible] an additional 10 ml. of the same benzene extract [illegible] thiobarbituric acid number; if not, a fresh benzene [illegible] and the determination performed as given by S[illegible] procedure was standardized with tetraethoxypropane [illegible] acid solution to malonaldehyde and enables the [illegible] as milligrams of malonaldehyde per 1,000 g. of [illegible] acid number.

c. Re[illegible]: [illegible] of these determinations run on the various [illegible] are shown in Table XV. The peroxide number of [illegible] about 20. The low moisture cereal bars, the potato [illegible] bars with acetyl glyceride coatings were the only [illegible] an appreciable amount of peroxide was found. The [illegible] of the different beef bars ranged from 2.3 to 3.3 µM/g. [illegible] bars had the highest carbonyl content, 3.64 µM/g. [illegible] The date, cereal, pea, and nonfat milk solids [illegible] carbonyl or less than 1 µM/g. The thiobarbituric [illegible] low for all the bar types.

K[illegible] that the Henick, Benca, and Mitchell carbonyl pro[illegible] for the saturated carbonyl component of [illegible] W[illegible] some recovery trials in which known amounts [illegible] to the beef samples. Recoveries were about 150 [illegible]. No trials were made with crotonaldehyde [illegible] of the butyr- and crotonaldehyde 2,4-D's [illegible] either the saturated or unsaturated component [illegible] method failed to give accurate

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* B[illegible] Bausch and Lomb Optical Company, Ro[illegible]

TABLE XV

PEROXIDE NUMBER, TOTAL CARBONYL CONTENT AND THIOBARBITURIC ACID NUMBER OF COMPRESSED FOOD BARS

Food Bar	Peroxide No. meq/1,000 g.*	Total Carbonyl umol/gram*	Thiobarbituric Acid No.**
Beef, low moisture	17.6 16.7-17.7***	3.30 3.10-3.50	0.16 0.14-0.19
Beef, high moisture	19.7 17.7-19.5	2.30 2.23-2.40	0.12 0.10-0.15
Beef, w/sorbitol	24.4 23.2-25.1	2.66 2.53-2.82	0.15 0.14-0.18
Beef, w/mono-, diglycerides	22.5 21.4-23.2	3.18 2.96-3.39	0.20 0.17-0.25
Dates, low moisture	0.0 -	0.21 0.18-0.25	0.01 0.00-0.02
Dates, high moisture	0.0 -	0.20 0.15-0.30	0.03 0.02-0.03
Dates, w/acetyl glyceride coating	2.2 -	0.35 0.32-0.39	0.05 0.02-0.13
Peas, low moisture	< 1.0 -		
Peas, high moisture	< 1.0 -		
Cereal, low moisture	3.4 3.1-4.1	0.44 0.44-0.45	[illegible] -
Cereal, high moisture	0.0 -	0.52 0.39-0.82	0.12 0.10-0.13
Nonfat milk solids	< 1.0	None detected	None detected
Potato chips	1.6 0.9-1.9	3.64 3.45-3.94	0.15 0.12-0.18

* Dry matter basis.
** Mg. malonaldehyde/1,000 g. dry matter.
*** Range of values.

differentiation between the concentration of butyraldehyde and crotonaldehyde, but the total carbonyl was within 5 per cent of the correct value. We recommend that the accuracy of the method should be checked for each individual food type for each new application. The method is simple and fairly rapid, and would be of definite value if it should prove to give good results for total carbonyl, even though it might not accurately differentiate between the concentrations of saturated and unsaturated carbonyls.

The thiobarbituric acid number (TBA) determination would appear to be of little usefulness since the results were so low. The results may have been a result of inadequate extraction. Kwan and Watts[25] suggest that water is an adequate solvent for preformed malonaldehyde in foods. The procedure of Sidwell et al. may not extract all the malonaldehyde from the benzene solution. Torladgis[26] used a distillation method. The optimum method of extraction should be investigated because the reaction of oxidized fats with thiobarbituric acid has been applied to the evaluation of a number of different products, a few of which are fish oils (deKoning and Silk[27]), fats and oils (Jacobson, Kirkpatrick, and Goff[28]) and raw meats (Keskinel, Ayres, and Snyder[29]).

9. Quantitative Changes Associated with Browning Reaction; Reducing Sugar and Amino Nitrogen Content

a. Method: The reducing sugar determination is discussed in Section II, B-3, on pp. 31 and 32. Amino nitrogen was determined on a water slurry of the food bar by the Van Slyke method.[30] The only modification of the procedure of the AOAC was to use, instead of a water extract, a water slurry, prepared by homogenizing in a Waring Blendor, a food bar or its equivalent in 100 ml. of distilled water. This homogenization facilitates the determination of amino nitrogen from the water-insoluble protein as well as from the water-soluble components. An aqueous solution of glycine was used as a standard. Recovery of amino nitrogen from glycine added to beef samples was within 5 per cent of the calculated value.

b. Results: Table XVI is a tabulation of the amino nitrogen content of the various test food bars. The beef bars contained an average of 5.2 to 5.7 mg/g. The pea, nonfat milk solids and potato chip bars contained between 4 and 5 mg/g, while the cereal and date bars contained less than 1 mg/g. The changes with storage are discussed in Section III.

TABLE XVI

AMINO NITROGEN CONTENT OF COMPRESSED FOOD BARS

Food Bar	Amino Nitrogen, mg/g*	
	Avg. Value	Range
Beef, low moisture	5.50	5.10-5.59
Beef, high moisture	5.60	4.68-6.07
Beef, w/sorbitol	5.18	4.71-5.71
Beef, w/mono-, diglycerides	5.73	5.03-6.43
Dates, low moisture	0.13	0.09-0.15
Dates, high moisture	0.18	0.13-0.31
Dates, w/acetyl glyceride coating	0.43	0.36-0.47
Peas, low moisture	4.13	3.35-4.90
Peas, high moisture	4.31	3.89-5.03
Cereal, low moisture	0.35	0.27-0.40
Cereal, high moisture	0.37	0.30-0.43
Nonfat milk solids	4.10	3.63-4.43
Potato chips	4.76	4.51-5.13

* Dry matter basis.

C. Microbiological Determinations

1. Total Aerobic Plate Counts

a. Method: Total aerobic plate counts, coliform plate counts and anaerobic spore counts were made according to procedures in "Recommended Methods for the Microbiological Examination of Foods."[31] Three 1:10 dilutions were prepared for each type of food bar, using five bars for each 1:10 slurry. Five plates were poured for each slurry at dilutions of 1:100, 1:1,000, and 1:10,000 for the aerobic plate counts. The reported count was based on the dilution which gave the most reliable count per plate.

b. Results: The microbiological counts are summarized in Table XVII. The total aerobic plate count for the beef bars was about 100/g, and the next highest were the cereal bars with 500-700/g. The aerobic count on the date bars was considerably greater, 5,000-6,000, for the high and low moisture groups and particularly those with acetyl glyceride coatings, about 23,000. A high proportion of the colonies were mold and yeast. The pea bars had a rather high count, averaging 12,000-20,000/g, while the nonfat milk solids and potato chip bars averaged 3,000-4,000/g. The higher moisture group of the date, pea, and cereal bars had higher counts than did the lower moisture group.

2. Coliform and Anaerobic Spore Count

a. Method: Five plates were poured for each slurry at dilutions of 1:10 and 1:100 for the coliform and anaerobic spore counts. We found that an evacuated desiccator or Brewer's Jar was better for the anaerobic spore count than trying to maintain anaerobic conditions by overlaying with anaerobic agar. No difficulties were experienced with these prescribed procedures.

b. Results: The coliform counts were 10 or less per gram for all but the nonfat milk solids bars which averaged 80/g. The anaerobic spore counts were also low except for the high moisture dates and the nonfat milk solids.

The effect of storage on the bacteriology of the compressed food bars is discussed in Section III.

TABLE XVII

THE BACTERIOLOGY OF COMPRESSED FOOD BARS

Food Bar	Total Aerobic Count (number/gram)	Coliform Count (number/gram)	Anaerobic Spore Count (number/gram)
Beef, low moisture	0.1*	0	0
	0-2.0**	-	-
Beef, high moisture	0.1	8	13
	0-0.2	0-14	4-20
Beef, w/3% sorbitol	0.1	3	0
	0-0.1	0-30	-
Beef, w/mono-, diglycerides	0.1	0	0
	0-0.1	-	-
Dates, low moisture	5.5	0	5
	3.4-8.4	-	0-20
Dates, high moisture	6.5***	3	38
	2.5-9.6	0-10	0-70
Dates, w/acetyl glyceride coating	13.6***	0	3
	12.2-15.2	-	0-20
Peas, low moisture	11.7	3	9
	7.3-20.1	0-10	0-40
Peas, high moisture	20.1	3	0
	13.0-33.3	0-10	-
Cereal, low moisture	0.5	0	5
	0.3-1.0	-	0-20
Cereal, high moisture	0.7	3	4
	0.2-1.3	0-10	0-20
Nonfat milk solids	3.2	80	55
	1.6-5.7	20-190	10-110
Potato chips	3.7	0	0
	2.0-5.7	-	-

* Times 10^3.

** Range from three slurries, three plates per slurry.

*** High percentage of yeast.

III. THE EFFECT OF STORAGE ON COMPRESSED FOOD BAR CHARACTERISTICS

A. Method

Approximately 200 bars of each variety were packaged and stored under four different conditions for three months, after which they were checked for the parameters discussed in Section II. The packaging and storage conditions were as follows:

1. Air-packed in sealed can, stored at 100°F.

2. Vacuum-packed, stored at 100°F.

3. Air-packed in sealed can, cycled between 0° and 40°F, three cycles per week.

4. Stored at 70°F, exposed to air at 40 per cent R.H.

The bars were packed about 40 each in 1-lb. coffee cans. The vacuum-packed were evacuated to 29 in. of mercury vacuum to attain a pO_2 of 1 mm. or less. The actual oxygen tension was not determined. At the barometric pressures encountered, the pO_2 of the air-packed cans came within the range of 135 - 145 mm. Hg. The pea bars were packaged in foil laminate pouches, seven to eight bars per pouch.

After three months in storage, the bars were tested by the procedures outlined in Section II. At least three bars from each storage condition were tested for the various physical characteristics. For most of the chemical determinations and water activity, four to five bars from separate cans were crushed and mixed and an aliquot removed for analysis. Five bars were used to prepare one slurry from each storage condition for the bacterial counts.

B. Results

The results of tests on the stored bars are summarized in Appendix A. There were no major changes in the apparent true density or apparent porosity of the food bars under any of the storage conditions.

imposed, Tables A-I and A-II. The porosity apparatus, which was broken before the beef bars were removed from storage was not repaired because its inaccuracy made it not worth the trouble. Therefore, data for the stored beef bars were not obtained.

The uncoated high moisture dates, and the high moisture dates coated with acetyl glyceride, when stored at 100°F, became very soft. This was reflected in the lowered hardness and shear values as shown in Tables A-III and A-IV. The shear and hardness values of these same bars were increased by three months' storage cycling between 0° and 40°F. Other than some small changes in the pea bars, the 100°F and 0° - 40°F cycling storage had had little effect on the hardness and shear values of the various test bars. Exposure to air and 40 per cent R.H. at 70°F tended to increase the hardness and shear of the beef bars. This effect seemed to be in the meat fibers rather than the over-all bar structure. The high moisture date bars lost moisture to the 40 per cent R.H. atmosphere, and their hardness and shear values tended to increase with storage while the values for the pea bars tended to decrease. The hardness values of the cereal bars were increased with storage under these conditions, but shear values were not affected. The potato chip bars were unaffected by the exposure to air at 40 per cent R.H., while the shear value of nonfat milk solids bars was increased.

The impact resistance, Table A-V, of the beef bars, cereal bars, and the potato chip bars was unaffected by any of the storage conditions. The value for pea bars stored, either air- or vacuum-packed, at 100°F or cycling between 0° - 40°F, was lower than the initial value, but exposure to air and 40 per cent R.H. at 70°F had no effect. Although the change was small, the impact resistance of nonfat milk solids bars exposed three months to air and 40 per cent R.H. was greater than either the initial value or the values for the other three storage conditions.

The test bars made from nonfat milk solids were not tested for dimensional stability after storage. With the exception of the cereal bars and the date bars, exposure to air and 40 per cent R.H. for three months decreased the dimensional stability at 100°F of the compressed food bars, Table A-VI-A. All date bars under these conditions and the low moisture date bars, when stored at 100°F and cycled between 0° and 40°F, had greater resistance to deformation than when first prepared. The high moisture date bars exhibited less dimensional stability when stored under the latter two conditions. The three months' storage at 100°F and cycling between 0° and 40°F did not noticeably change the dimensional stability of the beef, pea, cereal, or potato chip bars. Only the date bars were tested for dimensional stability at 40°F, Table A-VI-B. The 100°F storage had a definite detrimental effect on the two high moisture bars, but had no effect on the low moisture bars when they were subjected to 5 psi for 24 hr. at 40°F. Dimensional stability was good at 40°F for all the date bars stored under the 0° - 40°F temperature cycling and in air at 40 per cent R.H.

The effects of storage on cohesiveness are summarized in Table A-VII. No values were obtained on the stored beef, cereal or potato chip bars for cohesiveness because holes could not be drilled for mounting without the bar cracking or falling apart. The high moisture date bars were adversely affected by the 100°F storage, but the low moisture date bars were not. All the date bars had higher cohesiveness values when stored under temperature cycling and the loss of moisture to the 40 per cent R.H. atmosphere caused the high-moisture date bars to improve in cohesiveness. The fresh, low moisture pea bars had a 4.9 cohesiveness value, but exposure to 40 per cent R.H. dropped the value to 0; otherwise, storage had no effect on their cohesiveness. The pressure exerted on the evacuated foil laminate pouches in which the pea bars were packaged caused the high moisture pea bars to have a good cohesiveness value after three months at 100°F. If they had been packaged in cans, the value probably would have been 0 or near 0. The nonfat milk solids bars showed little change after storage, but there was some increase in cohesiveness by bars exposed to the 40 per cent R.H.

The water dispersion time for beef bars was unchanged by storage (Table A-VIII). Storage condition had no consistent effect on the dispersion time for date and cereal bars. Only 1 min. was required for dispersion of pea bars exposed to air and 40 per cent R.H., compared to initial values of 23 and 6 min., respectively, for low and high moisture pea bars. The other storage conditions had little effect on the dispersibility of the low moisture pea bars, but the consolidating effect of the external pressure on the foil laminate pouches was reflected in the increase in dispersion for the 100°F vacuum-packed, high moisture pea bars. Storage, especially the exposure to 40 per cent R.H., increased the dispersion time for the nonfat milk solids bars.

Although the wet and dry bulb temperatures in the constant temperature-humidity storage cabinet indicated a relative humidity of 40 - 45 per cent, the results shown in Table A-IX indicate that the chamber was at about 50 - 55 per cent R.H. The water activity of all the bars stored in the chamber, except the low moisture beef bars, ranged from 0.49 to 0.60. The water activity of the bars stored under the other three storage conditions in sealed containers did not differ appreciably from the value for the freshly prepared bars.

Permeability times, summarized in Table A-X, were not affected by storage with the exception of one group of the high moisture peas and the nonfat milk solids. The increase in permeability time for the vacuum-packed high moisture peas stored at 100°F is undoubtedly due to the consolidating effect of external air pressure on the flexible pack and consequently on the bars, themselves. There was some variation in the times for the nonfat milk solids bars, but these general conclusions can be made: storage at 100°F had little influence; the alternate freezing and thawing evidently caused some localized condensation and consequently plugging of pores so that the permeability time was longer after three months than for the fresh bar. Exposure to air and 40 per cent R.H. lowered the permeability time slightly.

Table A-XI shows the effect of storage on moisture content of the various food bars. The slight difference between the initial moisture values and the values for the bars stored in closed containers, both groups stored at 100°F, and the cycling temperature group, are undoubtedly due in part to sampling as well as analytical variability. However, in no case is the difference sufficient to warrant a conclusion that storage had any significant effect on moisture content. The moisture level of the bars stored in the constant temperature-humidity cabinet could have been predicted from the equilibrium relative humidity curves, Figs. 5 - 10. The actual moisture values indicate, as did the values for water activity, that the relative humidity was slightly greater than the 40 - 45 per cent indicated by the temperature sensors.

The free and bound lipid values for the stored bars are given in Tables A-XII and A-XIII. The fat analysis of dates without acetyl glyceride coatings and of the nonfat milk solids was not repeated after the three months' storage. There were no changes in free lipid content due to storage. The apparent bound lipid content of the pea, cereal, and potato chip bars did not change, but there were some definite changes in some of the beef bars. There was a tendency for the bound lipid level of the beef bars to be higher after the three months' storage. Whether these higher results were due to a change in some component, thus rendering it soluble in the chloroform:methanol solvent, is not known. These results indicate that determining total lipid or weight loss after simple chloroform:methanol is not a reliable method.

The reducing sugar content of bars stored at 100°F (see Table A-XIV) lessened during storage. This trend is particularly noticeable in the date bars, the greatest changes being in the high moisture groups. The reducing sugar content of the nonfat milk solids bars also showed a definite decrease when they were stored at 100°F. Air-packed bars cycled between 0° and 40°F underwent no change in reducing sugar content. Storage in air and 40 per cent R.H. generally had a lowering effect on reducing sugar content, but the effect was not as pronounced as with the 100°F storage.

Storage had no effect on the pH of a water slurry of the various food bars, Table A-XV, with the exception of the high moisture cereal bars. Although taste testing was not a requirement of the contract, most of the bars were so tasted. The high moisture cereal bars had a definite soapy taste following storage. Hydrolytic changes in the fat had evidently occurred and was reflected in the lowered pH of the water slurry, particularly for those bars stored at 100°F.

The effect of three months storage on the water-soluble fraction is summarized in Table A-XVI. The water-soluble fraction of the beef and pea bars was unaffected by storage. The date bars coated with acetyl glycerides were reduced slightly in their water solubility by the storage period but the uncoated date bars were unaffected. The cereal bars and the nonfat milk solids bars also had slightly lowered water-soluble fractions after the storage period while that of the potato chip bars was about 5 per cent greater than initially.

The equilibrium relative humidity data are presented in tabular form in Table A-XVII. With the exception of the bars stored in air and exposed to 40 per cent R.H., the equilibrium moisture content at the various relative humidities was essentially unaffected by storage. An imperfect hysteresis is seen in some of the bars exposed to the 40 per cent R.H. The beef bars thus stored lost moisture to the atmospheres of 42.8 per cent or lower relative humidity. They tended to come to equilibrium at slightly higher moisture levels than did the fresh bars; however, at the 80.7 per cent R.H. the equilibrium moisture contents were about the same as for the fresh bars. Also, the nonfat milk solids bars stored exposed to air and 40 per cent R.H., tended to reach slightly lower moisture levels than initially except at the high relative humidity. The other bars thus stored showed little change from the fresh condition. Complete sets of data were not determined for the date bars because of the long time needed. The effect of storage on the rate of equilibration to 40 per cent R.H. was not checked.

Thiobarbituric acid numbers were not determined for any of the stored bars, but peroxide number, Table A-XIII, and carbonyl content, Table A-XIX, were determined as quantitative aspects of oxygen uptake. The freshly prepared beef bars had peroxide numbers of about 20. These numbers decreased during the three months to about one-fourth to one-half the original value. The peroxide number of the stored cereal bars and potato chip bars was very low. Dates, peas, and nonfat milk solids were not analyzed for peroxide number. Carbonyl content was determined only on the beef, cereal and potato chip bars. The values for all these bars were somewhat variable but in no case was there a very large deviation from the initial values obtained on the freshly prepared bars.

The effect of storage on reducing sugar content was presented in Table A-XIV. The other factor measured incident to browning reaction was the amino nitrogen content, which is summarized in Table A-XX. There was little change with storage of the amino nitrogen content of the beef bars, except the level in the bars stored in air and exposed to 40 per cent R.H. was slightly lower than in the fresh bars. The date bars showed an over-all trend to higher values after storage, although the acetyl glyceride coated group was a little lower than the initial value. The amino nitrogen content of the stored pea bars was almost twice the initial values, whereas the concentration in the nonfat milk solids bars was about two-thirds the initial value. The amino nitrogen content of the potato chip bars was slightly lower following the storage period. Subjectively the date, pea, cereal, and nonfat milk solids bars had undergone visible browning in the 100°F storage groups. Little change in color could be seen for those bars stored in the air or under conditions of temperature cycling. Calculation of the molar change in reducing sugar and amino nitrogen for some of the bars shows that 1 mg. of amino nitrogen is equivalent to 7×10^{-5} mole and 1 per cent reducing sugar is equivalent to 6×10^{-5} mole/g. Thus, for nonfat milk solids air-packed and stored at 100°F, there was a reduction in reducing sugar of 18.6×10^{-5} moles and in amino nitrogen of 12.5×10^{-5} moles. These results are in good agreement with the concept that there is a condensation of sugar and amino nitrogen during nonenzymatic browning.

The effects of storage on the bacteriological picture of the compressed food bars are summarized in Tables A-XXI, A-XXII, and A-XXIII. With the notable exception of the low moisture peas and the potato chip bars, the total aerobic plate count of most of the bars stored for three months at 100°F was lower than the count on the fresh bars. The high counts for the high moisture beef, pea, and cereal bars stored with cycling temperature indicate there may have been some freezing and thawing with sufficient condensation at times to permit some bacterial growth. With the exception of the low moisture dates and the nonfat milk solids bars, the counts for the groups exposed to air and 40 per cent R.H. were lower than the initial counts. The covering of one of the four nonfat milk solids cans was touching the top layer of bars, and had evidently promoted localized moisture transfer and resultant mold growth. No other incidence of mold was noted for the samples stored in the constant temperature humidity cabinet. The initial coliform count was low for all bars but the nonfat milk solids; consequently storage under all conditions generally had no effect on the coliform count. The high moisture beef stored under temperature cycling did have a high coliform count as did the low moisture dates stored under the same conditions, again an indication that conditions suitable to support bacterial growth may have occurred.

The coliform counts for the nonfat milk solids fell some but to not less than one-fourth of the original count. The nonfat milk solids had a high anaerobic spore count after storage when compared to other bars. This count is a reflection of the high initial count, as most of the other bars had low initial counts and either very low or 0 after storage.

The oxygen uptake of the bars is given in Table A-XXIV. The beef, acetyl glyceride coated dates, cereal, and potato chip bars had a slower rate of oxygen uptake; consequently a lower total uptake, after the three months' storage than did the freshly prepared bars. The rate of uptake by the stored pea and milk bars was about the same as the fresh bars. Samples of high and low moisture date bars from all the storage conditions were not run, but the rate of uptake was greater than initially for all the samples tested. The bars stored exposed to air and 40 per cent R.H., generally had a higher rate of uptake than did the rest of the stored bars, particularly the beef bars and the nonfat milk solids bars. Bacteria may have contributed to this result.

IV. PREPARATION OF TEST-COMPRESSED FOOD BARS

Following is a brief description of the preparation of each type of food bar. A list of the individual ingredients and their sources is given in Appendix B.

The final mixes were tableted in a Stokes R-1 press. The die used yielded a food bar 1-1/4 in. square. A depth of fill was used to yield bars about 1/2 in. thick.

A. Cooked, Freeze-Dried Ground Beef

The dry ingredients of the beef bars consisted of 75 per cent by weight of cooked, freeze-dried ground beef, and 25 per cent by weight of dehydrated chicken broth. The beef was placed in a mixer and moistened by the addition of 30 cc. of water per 100 g. of total dry ingredients. The water was added slowly with mixing and the batch mixed for several minutes after the addition of all the water. The dehydrated chicken broth was then added, in small increments and with mixing. The final mix was placed in a closed container and allowed to temper overnight, then tableted. The addition of sorbitol and mono- and diglycerides was made at the expense of the beef. After tableting, the bars were placed in a forced air drier and dried to the desired moisture level. The drier temperature was 110 ± 2°F.

Physically these bars were very porous and quite fragile.

B. Date Bars

As purchased, the moisture level of the dates was intermediate between the two desired levels. The dates were torn apart, placed on large trays and sprayed with a calculated amount of water to attain the high moisture level. The low moisture level was attained by drying in a forced draft oven. The dates, with the moisture level adjusted, were ground in a meat grinder through 1/8-in. plate, then rolled out in sheets 1/2 in. thick. The sheets were scored and cut out by hand. The bars which required acetyl glyceride coating were dipped in a mixture of acetyl glyceride held at a temperature of about 70°C. Excess coating was allowed to run off and the bars set aside at room temperature to allow the coating to harden. The acetyl glyceride mixture consisted of 10 per cent Myvacet 9-40 and 90 per cent Myvacet 5-00.

C. Cooked, Freeze-Dried Pea Bars

The cooked, freeze-dried peas were ground in a Wiley mill. The ground peas and sorbitol were mixed at the weight ratio of 90:10, respectively. Water was then added to the dry mix at the rate of 10 g/100 g of dry material, and the moistened mix tableted. Teflon tape had to be used on the punches because the mix stuck to them. The pea bars were not entirely satisfactory in that there was delamination when dried.

D. Cereal Bars

Composition of the cereal bars was as follows: ground corn flakes, 62 per cent; nonfat dry milk, 14 per cent; shortening, 10 per cent; and sugar, 14 per cent. The ingredients were mixed and then moistened just prior to tableting. Since a moisture level of 8 - 10 per cent was difficult to handle in the press, one batch was moistened to about 12 per cent level and another to about 6 per cent and the resulting bars dried to the desired moisture levels. These bars were crumbly and difficult to handle.

E. Nonfat Milk Solids Bars

The freshly opened nonfat milk solids were mixed with 0.5 per cent by weight of magnesium stearate and tableted. The magnesium stearate served as a release agent. The bars tended to hang in the die during the ejection step and would split badly when the release agent was used.

F. Potato Chip Bars

Commercially available potato chips were ground to a particle size of 1/8 in. or less in a Labconco laboratory mill. Dehydrated chicken broth was mixed with the ground potato chips at a level of 10 per cent by weight and then water at the rate of 5 g/100 g of mixture. The moistened mixture was then tableted on the Stokes R-1 press at a pressure which would give a compact bar but a minimum of fat loss. They were then dried about 4 hr. at 100°F in a forced draft oven, allowed to cool and packaged.

V. CONCLUSIONS AND RECOMMENDATIONS

Satisfactory methods are outlined and were used to quantitatively measure the following characteristics of compressed dehydrated food bars: (a) hardness; (b) shear; (c) impact resistance; (d) dimensional stability; (e) cohesiveness; (f) dispersibility in water; (g) water activity; (h) permeability to gas; (i) moisture content; (j) free lipid content; (k) pH; (l) water-soluble fraction; (m) equilibrium relative humidity curve; and (n) rate of equilibration to 40 per cent R.H. The methods used for surveying the bacteriology of the food bars, specifically the total aerobic plate count, coliform count, and anaerobic spore count, were also found to be reliable. The results were found to be reproducible when the tests were performed in the prescribed manner by different technicians.

The determination of bulk density as performed in this project was satisfactory, but accurate true densities could not be determined because of incorrect results for porosity. We recommend that porosity and true density be calculated in conjunction with the use of the air comparison pycnometer to determine the true volume of solids. The procedure outlined herein for determining stickiness with the Du Nouy tensiometer may be too sensitive. A series of foods should be rated by both a test panel and the tensiometer to see if the method seems useful. The food bars were found to be inelastic and only for certain types of compressed food bars does it seem that elasticity is applicable.

A chloroform:methanol solution, 2:1 volume ratio, was found to extract nonlipid materials from the food materials tested. Consequently, bound lipid values calculated by subtracting the free fat from the total fat, which was taken as the weight loss due to chloroform: methanol extraction, were inaccurate. The modification suggested of extracting the chloroform:methanol extract with water to remove water-soluble components and subsequent evaporation of the organic solvent and weighing of the lipid residue should prove to give satisfactory results. The accuracy of using the outlined method of determining reducing sugars present in small concentrations in foods should be further investigated. The method used gave readily duplicable results for nonfat milk solids and dates, both in replicate samples by the same technician and by different technicians.

Measuring oxygen uptake by means of the Warburg respirometer was satisfactory under the conditions employed. However, the method should be correlated with conditions normally encountered, such as atmospheric oxygen tension instead of nearly pure oxygen and other temperatures than 50°C. The three determinations performed as indices of chemical change due to oxygen uptake, peroxide number, carbonyl content, and thiobarbituric acid number, should also be correlated with the amount of oxygen uptake. For reasons pointed out in the discussion of the respective determinations, the accuracy of the carbonyl and thiobarbituric acid determinations should also be further investigated. There may be other indices of change incident to oxygen uptake than the ones we have employed. The relation of oxygen uptake to stability of foods needs more thorough investigation.

The determination of amino nitrogen by the Van Slyke manometric method was found to be reliable, and results were easily duplicated by different operators after some practice with the machine. However, changes in amino nitrogen and reducing sugar contents were not easily correlated in relation to browning. The analysis of a larger number of samples may have aided in this respect.

Three months' storage of the test bars did not result in any great changes in most of the parameters studied. Density and porosity were essentially unaffected by any of the storage conditions. Except for the date bars, hardness, shear, impact resistance, cohesiveness, and permeability were practically unaffected by the storage condition used. The date bars tended to soften considerably when stored at 100°F. The three months' exposure to air and 40 per cent R.H. did produce some noticeable changes in the values for these characteristics in some of the food bars. Water dispersibility was not changed appreciably by storage, nor was water activity for those bars sealed from the atmosphere. Bars exposed to the 40 per cent R.H. equilibrated to a water activity and moisture content compatible with that level of relative humidity. The moisture content of the bars in the sealed containers did not change with storage. Of the chemical characteristics studied, some small changes were noted in some instances in reducing sugar, pH, and amino nitrogen content. Some noticeable browning had occurred in dates, peas, nonfat milk solids, and cereal bars which could be related to changes in reducing sugar and amino nitrogen content.

The bacterial population tended to decrease with storage. There were indications that storage with alternating freeze-thaw cycles may provide conditions suitable to promote bacterial growth.

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APPENDIX A

RESULTS OF OBJECTIVE MEASUREMENTS OF STORED COMPRESSED FOOD BARS

TABLE A-I

APPARENT TRUE DENSITY MEASUREMENTS OF COMPRESSED FOOD BARS STORED FOR THREE MONTHS

(grams per cubic centimeter)

Food Bar	Fresh	Packaging and Three-Month Storage Condition: Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Dates, low moisture	1.25	1.22	0.89	1.22	1.17
Dates, high moisture	1.23	1.20	1.[illegible]	1.15	1.20
Dates, w/acetyl glyceride coating	1.20	-	-	-	-
Peas, low moisture	1.04	1.08	1.06	1.02	1.01
Peas, high moisture	1.00	0.95	1.04	0.90	0.93
Cereal, low moisture	1.19	1.18	1.22	1.21	1.19
Cereal, high moisture	1.16	1.16	1.16	1.15	1.16
Nonfat milk solids	1.33	1.34	1.32	1.33	1.31
Potato chips	0.89	0.84	0.86	0.84	0.88

TABLE A-51

APPARENT POROSITY MEASUREMENTS OF COMPRESSED FOOD BARS STORED FOR THREE MONTHS

(pore volume as per cent of bulk volume)

	Packaging and Three-Month Storage Condition				
Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Dates, low moisture	3	6	4	3	3
Dates, high moisture	5	6	4	3	5
Dates, w/acetyl glyceride coating	5	-	-	-	-
Peas, low moisture	35	36	35	35	32
Peas, high moisture	33	34	31	33	32
Cereal, low moisture	24	25	26	27	27
Cereal, high moisture	24	24	24	27	23
Nonfat milk solids	21	21	21	21	16
Potato chips	21	22	24	21	23

TABLE A-III

HARDNESS MEASUREMENTS (POUNDS) OF COMPRESSED FOOD BARS STORED FOR THREE MONTHS

Food Bar	Packaging and Three-Month Storage Condition				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	5	6	6	8	12
Beef, high moisture	5	6	5	5	13
Beef, w/sorbitol	4	4	4	4	8
Beef, w/mono-, diglycerides	5	4	4	4	9
Dates, low moisture	23	20	13	29	20
Dates, high moisture	4	< 1	0	10	9
Dates, w/acetyl glyceride coating	5	0	0	7	10
Peas, low moisture	61	> 70	> 70	57	61
Peas, high moisture	52	42	> 70	38	33
Cereal, low moisture	16	14	17	19	26
Cereal, high moisture	19	25	26	20	23
Nonfat milk solids	> 70	> 70	> 70	> 70	> 70
Potato chips	14	13	14	15	11

TABLE A-IV

EFFECT OF THREE-MONTHS' STORAGE ON SHEAR VALUES (POUNDS)
OF COMPRESSED FOOD BARS

Food Bar	Fresh	Packaging and Three-Month Storage Condition: Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	3	3	4	2	5
Beef, high moisture	3	2	4	3	7
Beef, w/sorbitol	2	2	1	2	8
Beef, w/mono-, diglycerides	2	2	2	2	4
Dates, low moisture	27	24	18	35	21
Dates, high moisture	5	1	1	14	13
Dates, w/acetyl glyceride coating	6	1	0	8	12
Peas, low moisture	41	39	43	36	27
Peas, high moisture	38	25	43	25	22
Cereal, low moisture	6	5	6	9	6
Cereal, high moisture	8	12	12	6	8
Nonfat milk solids	42	52	40	60	53
Potato chips	10	10	12	8	7

TABLE A-V

EFFECT OF THREE-MONTHS' STORAGE ON THE IMPACT RESISTANCE OF COMPRESSED FOOD BARS

(inch-pounds of force to cause shattering)

	Packaging and Three-Month Storage Condition				
Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H.,70°F)
Beef, low moisture	0.5	0.5	0.5	0.5	1.0
Beef, high moisture	0.5	0.5	0.5	0.5	1.0
Beef, w/sorbitol	0.5	0.5	0.5	0.5	1.0
Beef, w/mono-, diglycerides	0.5	0.5	0.5	0.5	1.0
Peas, low moisture	9	4	4	5	13
Peas, high moisture	12	5	10	4	10
Cereal, low moisture	1.5	1.0	1.0	1.5	1.5
Cereal, high moisture	1.5	1.5	1.5	2.0	1.5
Nonfat milk solids	0.6	0.5	0.5	0.5	3
Potato chips	1.5	1.0	1.0	1.0	1.0

TABLE A-VI-A

EFFECT OF THREE-MONTHS' STORAGE ON THE DIMENSIONAL STABILITY OF COMPRESSED FOOD BARS

(per cent deformation under 5 psi for 24 hr. at 100°F)

	Packaging and Three-Month Storage Condition				
Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	< 1*	1	1	***	4
	5**	7	6	8	19
Beef, high moisture	3	4	3	4	12
	11	12	11	14	26
Beef, w/sorbitol	1	1	< 1	< 1	19
	5	5	6	4	30
Beef, w/mono-, diglycerides	< 1	1	< 1	2	7
	5	2	2	8	21
Dates, low moisture	49	26	18	2	17
	34	21	33	8	18
Dates, high moisture	146	167	168	156	110
	59	66	66	66	59
Dates, w/acetyl glyceride coating	157	196	251	173	99
	64	65	70	68	55
Peas, low moisture	< 1	***	***	***	3
	1	2	***	1	7
Peas, high moisture	< 1	2	< 1	3	5
	8	8	3	5	17
Cereal, low moisture	< 1	< 1	***	2	4
	2	2	4	2	4
Cereal, high moisture	14	4	4	9	4
	16	9	10	13	4
Nonfat milk solids	***	****	****	****	****
Potato chips	17	2	4	4	10
	12	9	10	13	21

* Per cent deformation in lateral dimension, upper figure.

** Per cent deformation in thickness, lower figure.

*** No change.

**** Not tested.

TABLE A-VI-B

EFFECT OF THREE-MONTHS' STORAGE ON THE DIMENSIONAL STABILITY OF COMPRESSED FOOD BARS

(per cent deformation under 5 psi for 24 hr. at 40°F)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition				
Dates, low moisture	4	*	*	< 1	*
	5	1	8	3	1
Dates, high moisture	4	39	29	3	< 1
	8	24	30	4	2
Dates, w/acetyl glyceride coating	2	16	96	2	1
	4	15	45	3	2

* No change.

TABLE A-VII

EFFECT OF STORAGE ON THE COHESIVENESS OF COMPRESSED FOOD BARS

(pounds of force to pull bars apart)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition				
Dates, low moisture	4.8	8.8	3.8	10.5	4.5
Dates, high moisture	0.0	0.0	0.0	3.3	2.5
Dates, w/acetyl glyceride coating	1.2	0.0	0.0	2.5	3.3
Peas, low moisture	4.9	3.0	4.6	5.1	0.0
Peas, high moisture	0.0	0.2	6.6	0.0	0.0
Nonfat milk solids	12.0	14.8	9.2	14.0	16.5

TABLE A-VIII

EFFECT OF THREE-MONTHS' STORAGE ON THE WATER DISPERSION TIME OF COMPRESSED FOOD BARS

(minutes for dispersion in 10 volumes of water, shaken at 140 ± 10 strokes/minute)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition				
Beef, low moisture	< 1	< 1	< 1	< 1	< 1
Beef, high moisture	< 1	< 1	< 1	< 1	< 1
Beef, w/sorbitol	< 1	< 1	< 1	< 1	< 1
Beef, w/mono-, diglycerides	1	3	4	4	1
Dates, low moisture	420	260	360	390	370
Dates, high moisture	273	240	240	280	290
Dates, w/acetyl glyceride coating	288	315	285	405	360
Peas. low moisture	23	15	20	15	1
Peas, high moisture	6	< 1	20	12	< 1
Cereal, low moisture	316	320	290	290	255
Cereal, high moisture	175	97	155	240	240
Nonfat milk solids	150	295	295	295	> 480
Potato chips	> 480	460	> 480	> 480	> 480

TABLE A-IX

EFFECT OF THREE-MONTHS' STORAGE ON THE WATER ACTIVITY OF COMPRESSED FOOD BARS

(activity at 25°C)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H.,70°F)
	Packaging and Three-Month Storage Condition				
Beef, low moisture	0.05	< 0.05	< 0.05	< 0.05	0.42
Beef, high moisture	0.26	0.30	0.25	0.25	0.53
Beef, w/sorbitol	0.06	0.08	0.07	0.06	0.49
Beef, w/mono-, di-glycerides	0.06	0.06	0.07	0.05	0.50
Dates, low moisture	0.40	0.46	0.47	0.44	0.49
Dates, high moisture	0.53	0.54	0.57	0.56	0.57
Dates, w/acetyl glyceride coating	0.57	0.54	0.56	0.58	0.60
Peas, low moisture	0.08	0.07	0.06	0.05	0.57
Peas, high moisture	0.36	0.41	0.41	0.38	0.55
Cereal, low moisture	0.18	0.24	0.21	0.18	0.55
Cereal, high moisture	0.62	0.66	0.66	0.67	0.54
Nonfat milk solids	0.12	0.14	0.15	0.17	0.56
Potato chips	0.40	0.44	0.44	0.43	0.48

TABLE A-X

EFFECT OF THREE-MONTHS' STORAGE ON PERMEABILITY TIME OF COMPRESSED FOOD BARS

(seconds required to draw 50 cc. of air through bars)

Food Bar	Packaging and Three-Month Storage Condition: Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	2.5	2.6	2.6	2.6	2.6
Beef, high moisture	2.3	2.5	2.6	2.5	2.8
Beef, w/sorbitol	2.4	2.8	2.6	2.8	2.8
Beef, w/mono-, diglycerides	2.5	2.8	2.7	3.8	2.6
Dates, low moisture	← Nonpermeable →				
Dates, high moisture	← Nonpermeable →				
Dates, w/acetyl glyceride coating	← Nonpermeable →				
Peas, low moisture	13.8	20.5	20.5	14.0	21.5
Peas, high moisture	14.7	10.0	58.7	12.2	17.2
Cereal, low moisture	3.6	3.5	3.6	3.5	4.0
Cereal, high moisture	3.9	3.0	3.0	3.0	3.0
Nonfat milk solids	671	630	606	720	571
Potato chips	2.4	2.5	2.5	2.8	2.6

TABLE A-XI

EFFECT OF THREE-MONTHS' STORAGE ON MOISTURE CONTENT OF COMPRESSED FOOD BARS

(per cent)

Food Bar	Packaging and Three-Month Storage Condition				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H.,70°F)
Beef, low moisture	2.1	1.8	1.8	1.9	9.0
	(2.5)*	(2.2)	(2.2)	(2.3)	(10.7)
Beef, high moisture	6.4	6.1	5.4	6.5	12.2
	(7.6)	(7.2)	(6.3)	(7.8)	(14.4)
Beef, w/sorbitol	1.6	1.2	1.1	1.2	10.3
	(1.9)	(1.4)	(1.3)	(1.4)	(12.4)
Beef, w/mono-, diglycerides	1.6	1.6	1.7	1.7	10.1
	(1.9)	(2.0)	(2.1)	(2.1)	(12.4)
Dates, low moisture	9.6	9.2	9.2	8.8	12.4
Dates, high moisture	15.4	14.4	14.6	14.4	15.1
Dates, w/acetyl glyceride coating	16.4	15.3	16.7	14.7	14.7
Peas, low moisture	2.4	2.3	2.3	2.4	11.2
Peas, high moisture	6.8	7.1	6.9	7.0	11.0
Cereal, low moisture	3.9	3.4	3.4	3.2	8.5
Cereal, high moisture	9.5	9.0	9.2	8.8	8.5
Nonfat milk solids	3.5	2.9	2.9	3.0	10.3
Potato chips	5.6	4.8	5.2	5.0	6.3

* Figure in parentheses on fat-free basis.

TABLE A-XII

EFFECT OF THREE-MONTHS' STORAGE ON FREE FAT CONTENT OF COMPRESSED FOOD BARS

(per cent of dry matter)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition				
Beef, low moisture	16.2	17.5	17.0	18.0	15.8
Beef, high moisture	16.2	16.8	16.1	17.2	15.4
Beef, w/sorbitol	16.8	15.7	15.6	15.5	17.1
Beef, w/mono-, diglycerides	17.6	18.1	17.5	17.6	18.3
Dates, w/acetyl glyceride coating	1.9	2.3	2.9	3.0	2.8
Peas, low moisture	2.9	2.4	2.6	2.4	3.0
Peas, high moisture	3.2	2.9	2.4	2.1	3.1
Cereal, low moisture	14.5	14.6	14.2	14.2	14.1
Cereal, high moisture	13.9	13.6	13.7	14.1	14.4
Potato chips	31.8	31.3	27.6	31.7	25.2

TABLE A-XIII

EFFECT OF THREE-MONTHS' STORAGE ON APPARENT BOUND LIPID CONTENT OF COMPRESSED FOOD BARS

(per cent of dry matter)

Food Bar	Packaging and Three-Month Storage Condition				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	4.4	6.3	9.0	8.7	9.2
Beef, high moisture	4.3	6.7	6.2	6.5	7.2
Beef, w/sorbitol	7.7	8.4	9.9	10.2	11.0
Beef, w/mono-, diglycerides	7.6	6.4	7.5	7.1	9.4
Peas, low moisture	15.0	12.3	9.7	14.6	12.6
Peas, high moisture	11.4	12.6	13.2	10.3	15.3
Cereal, low moisture	3.0	3.0	3.7	2.1	2.4
Cereal, high moisture	3.2	1.4	1.6	2.5	1.8
Potato chips	0.5	0.9	0.1	0.6	1.3

TABLE A-XIV

EFFECT OF THREE-MONTHS' STORAGE ON REDUCING SUGAR CONTENT OF COMPRESSED FOOD BARS

(per cent glucose equivalent on dry matter basis)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition				
Beef, low moisture	0.24	0.22	0.19	0.22	0.16
Beef, high moisture	0.30	0.25	0.24	0.22	0.22
Beef, w/sorbitol	0.15	0.31	0.25	0.24	0.23
Beef, w/mono-, diglycerides	0.20	0.26	0.27	0.24	0.25
Dates, low moisture	73.8	72.2	71.6	74.6	74.3
Dates, high moisture	77.5	67.8	65.8	76.2	73.6
Dates, w/acetyl glyceride coating	78.0	70.1	73.5	77.3	77.0
Peas, low moisture	0.23	0.16	0.30	0.35	-
Peas, high moisture	0.38	0.27	0.17	0.31	-
Cereal, low moisture	6.4	6.2	6.4	6.4	6.2
Cereal, high moisture	6.6	6.2	6.0	6.2	6.2
Nonfat milk solids	26.3	23.2	24.2	25.2	27.2
Potato chips	0.15	0.20	0.20	0.22	0.19

TABLE A-XV

EFFECT OF THREE-MONTHS' STORAGE ON THE pH OF A WATER SUSPENSION OF COMPRESSED FOOD BARS

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Packaging and Three-Month Storage Condition (pH)				
Beef, low moisture	5.7	5.7	5.7	5.7	5 7
Beef, high moisture	5.7	5.7	5.7	5.7	5.7
Beef, w/sorbitol	5.7	5.8	5.8	5.8	5.8
Beef, w/mono-, diglycerides	5.7	5.8	5.8	5.8	5.8
Dates, low moisture	5.8	5.6	5.5	5.8	5.8
Dates, high moisture	5.8	5.4	5.4	5.9	5.8
Dates, w/acetyl glyceride coating	5.6	5.5	5.5	5.8	5.9
Peas, low moisture	7.0	7.0	7.0	7.0	7.0
Peas, high moisture	7.0	7.0	7.0	7.0	7.0
Cereal, low moisture	6.4	6.2	6.2	6.4	6.4
Cereal, high moisture	6.4	5.9	6.0	6.2	6.2
Nonfat milk solids	6.5	6.5	6.5	6.5	6.5
Potato chips	5.8	5.8	5.8	5.9	5.9

TABLE A-XVI

EFFECT OF THREE-MONTHS' STORAGE ON THE WATER-SOLUBLE FRACTION OF COMPRESSED FOOD BARS

(per cent of dry matter)

Food Bar	Packaging and Three-Month Storage Condition				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	27	27	28	29	29
Beef, high moisture	27	28	27	26	26
Beef, w/sorbitol	29	29	29	28	26
Beef, w/mono-, diglycerides	29	29	27	27	28
Dates, low moisture	86	86	89	88	90
Dates, high moisture	89	86	86	87	88
Dates, w/acetyl glyceride coating	86	76	78	74	80
Peas, low moisture	41	42	41	41	39
Peas, high moisture	42	38	41	42	38
Cereal, low moisture	41	33	34	34	34
Cereal, high moisture	38	31	31	36	36
Nonfat milk solids	99	95	96	94	96
Potato chips	17	24	22	22	23

TABLE A-XVII

EFFECT OF THREE-MONTHS' STORAGE ON THE EQUILIBRIUM-RELATIVE HUMIDITY DATA OF COMPRESSED FOOD BARS

Atmosphere Relative Humidity (%)	Equilibrium Moisture Content, %				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Beef, low moisture				
7.0	2.2	2.2	2.2	2.4	3.2
11.0	2.9	3.1	3.1	3.2	3.7
22.4	4.6	4.9	4.8	4.8	5.4
42.8	8.2	8.6	8.5	8.5	8.8
80.7	29.1	28.5	28.5	29.1	28.6
	Beef, high moisture				
7.0	3.0	3.0	2.9	3.3	3.3
11.0	3.6	3.4	3.4	3.7	3.9
22.4	5.0	4.6	4.6	5.1	5.4
42.8	8.4	8.3	8.3	8.7	9.0
80.7	27.9	29.4	29.3	28.4	28.5
	Beef, w/sorbitol				
7.0	2.4	2.1	2.1	2.5	2.8
11.0	3.0	3.0	3.0	3.1	3.5
22.4	4.6	4.8	4.8	4.9	5.1
42.8	8.5	8.5	8.7	8.8	8.9
80.7	28.9	29.9	29.2	29.8	29.4
	Beef, w/mono-, diglycerides				
7.0	2.6	2.4	2.4	2.5	3.0
11.0	3.0	3.1	3.2	3.3	3.6
22.4	4.9	4.8	5.0	4.9	5.2
42.8	8.4	8.3	8.3	8.5	8.8
80.7	29.6	28.0	28.2	28.8	28.6

TABLE A-XVII (Continued)

Atmosphere Relative Humidity (%)	Equilibrium Moisture Content, %				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Dates, low moisture				
7.0	6.6				
11.0	6.7				
22.4	7.1				
42.8	8.8	8.6	8.5	8.6	8.4
80.7	28.1				
	Dates, high moisture				
7.0	7.9				
11.0	8.0				
22.4	7.9				
42.8	10.3	9.5	9.3	10.2	10.5
80.7	31.8				
	Dates, w/acetyl glyceride coating				
7.0	7.8				
11.0	7.8				
22.4	7.9	6.6	7.5	7.5	7.5
42.8	10.1	9.8	10.6	10.3	10.5
80.7	34.3	30.2	29.8	30.0	28.4
	Peas, low moisture				
7.0	2.6	2.2	2.1	2.3	2.6
11.0	3.0	2.4	2.4	2.7	3.1
22.4	4.2	3.8	3.8	4.0	4.2
42.8	7.2	7.1	7.1	7.1	7.4
80.7	18.9	21.5	21.2	21.4	22.3

TABLE A-XVII (Continued)

Atmosphere Relative Humidity (%)	Equilibrium Moisture Content, %				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
			Peas, high moisture		
7.0	3.2	2.7	2.7	2.9	2.7
11.0	3.6	3.2	3.1	3.3	3.1
22.4	4.6	4.1	4.1	4.3	4.2
42.8	7.2	6.9	6.9	7.0	7.3
80.7	19.4	23.2	21.6	21.8	21.9
			Cereal, low moisture		
7.0	3.0	2.7	2.7	2.8	3.0
11.0	3.4	3.2	3.2	3.3	3.6
22.4	4.2	3.9	3.8	4.1	4.6
42.8	5.9	5.7	5.7	5.8	6.3
80.7	14.3	15.1	14.6	14.6	14.7
			Cereal, high moisture		
7.0	3.1	3.0	3.0	3.0	3.0
11.0	3.6	3.4	3.4	3.5	3.5
22.4	4.7	4.4	4.4	4.5	4.5
42.8	6.5	6.1	6.1	6.2	6.2
80.7	13.4	14.5	14.5	14.7	15.0
			Nonfat milk solids		
7.0	3.2	2.7	2.7	2.8	2.8
11.0	3.3	2.9	2.9	3.1	3.3
22.4	4.7	3.5	3.5	3.9	4.1
42.8	7.4	7.0	6.9	7.0	5.6
80.7	11.8	12.8	13.0	12.9	12.1

TABLE A-XVII (Concluded)

Atmosphere Relative Humidity (%)	Equilibrium Moisture Content, % Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
	Potato chips				
7.0	2.6	2.4	2.5	2.3	2.6
11.0	2.9	2.7	2.8	2.6	3.0
22.4	3.9	3.6	3.8	3.6	4.2
42.8	5.5	4.8	5.2	5.1	5.9
80.7	15.2	16.5	17.6	16.5	17.3

TABLE A-XVIII

EFFECT OF THREE-MONTHS' STORAGE ON THE PEROXIDE NUMBER OF COMPRESSED FOOD BARS

(milliequivalents peroxide per 1,000 grams dry matter)

Food Bar	Fresh	Packaging and Three-Month Storage Condition Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	17.6	6.2	5.9	3.6	3.4
Beef, high moisture	19.7	4.4	6.0	8.2	9.6
Beef, w/sorbitol	24.4	5.1	9.5	5.3	6.8
Beef, w/mono-, diglycerides	22.5	6.0	5.3	6.1	6.9
Peas, low moisture	< 1.0	-	-	-	-
Peas, high moisture	< 1.0	-	-	-	-
Cereal, low moisture	3.4	1.4	2.0	0.8	0.0
Cereal, high moisture	0.0	0.0	0.0	0.0	0.0
Nonfat milk solids	< 1.0	-	-	-	-
Potato chips	1.6	0.0	0.0	1.0	1.5

TABLE A-XIX

EFFECT OF THREE-MONTHS' STORAGE ON THE CARBONYL CONTENT OF COMPRESSED FOOD BARS

(micromoles per gram of dry matter)

Food Bar	Fresh	Packaging and Three-Month Storage Condition: Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	3.30	2.49	2.55	2.46	2.66
Beef, high moisture	2.30	2.96	2.86	2.54	3.00
Beef, w/sorbitol	2.66	2.13	1.98	2.21	2.15
Beef, w/mono-, diglycerides	3.18	3.09	2.07	2.17	2.55
Cereal, low moisture	0.44	0.52	0.53	0.49	0.53
Cereal, high moisture	0.52	0.65	0.70	0.68	0.56
Potato chips	3.64	3.08	2.63	3.34	2.67

TABLE A-XX

EFFECT OF THREE-MONTHS' STORAGE ON THE AMINO NITROGEN CONTENT OF COMPRESSED FOOD BARS

(milligrams amino nitrogen per gram dry matter)

Food Bar	Fresh	Packaging and Three-Month Storage Condition: Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	5.50	4.59	4.90	5.55	4.98
Beef, high moisture	5.60	6.02	5.79	5.76	4.91
Beef, w/sorbitol	5.18	5.62	5.40	5.31	4.87
Beef, w/mono-, diglycerides	5.73	5.91	5.54	5.59	5.01
Dates, low moisture	0.13	0.21	0.23	0.19	0.37
Dates, high moisture	0.18	0.24	0.20	0.37	0.46
Dates, w/acetyl glyceride coating	0.43	0.20	0.19	0.31	0.29
Peas, low moisture	4.13	7.28	8.35	7.70	7.75
Peas, high moisture	4.31	7.40	7.36	7.61	7.68
Cereal, low moisture	0.35	0.27	0.26	0.27	0.31
Cereal, high moisture	0.37	0.22	0.26	0.43	0.32
Nonfat milk solids	4.10	2.31	2.37	2.51	2.40
Potato chips	4.76	3.85	3.96	3.96	4.12

TABLE A-XXI

EFFECT OF THREE-MONTHS' STORAGE ON TOTAL AEROBIC PLATE COUNT OF COMPRESSED FOOD BARS

(organisms per gram)

Food Bar	Packaging and Three-Month Storage Condition				
	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	< 100	< 100	< 100	< 100	< 100
Beef, high moisture	100	200	< 100	1,700	100
Beef, w/sorbitol	100	< 100	100	< 100	< 100
Beef, w/mono-, diglycerides	< 100	100	< 100	< 100	100
Dates, low moisture	5,500	2,000	3,700	6,400	11,300
Dates, high moisture	6,500	4,300	3,600	3,700	2,400
Dates, w/acetyl glyceride coating	13,600	400	500	4,600	6,600
Peas, low moisture	11,700	14,000	12,300	12,900	4,700
Peas, hign moisture	20,100	2,400	3,100	13,400	4,700
Cereal, low moisture	500	200	200	200	200
Cereal, high moisture	700	300	900	1,300	400
Nonfat milk solids	3,200	700	700	600	3,900
Potato chips	3,700	7,000	2,300	300	100

TABLE A-XXII

EFFECT OF THREE-MONTHS' STORAGE ON THE COLIFORM COUNT OF COMPRESSED FOOD BARS

(organisms per gram)

Food Bar	Fresh	Packaging and Three-Month Storage Condition Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	0	0	0	0	0
Beef, high moisture	8	0	0	900	0
Beef, w/sorbitol	3	0	0	0	0
Beef, w/mono-, diglycerides	0	0	0	0	0
Dates, low moisture	0	0	2	50	0
Dates, high moisture	3	0	0	4	0
Dates, w/acetyl glyceride coating	0	0	0	0	0
Peas, low moisture	3	2	2	10	0
Peas, high moisture	3	0	0	0	0
Cereal, low moisture	0	0	0	0	0
Cereal, high moisture	3	0	0	0	0
Nonfat milk solids	80	40	30	20	20
Potato chips	0	0	0	0	0

TABLE A-XXIII

EFFECT OF THREE-MONTHS' STORAGE ON THE ANAEROBIC SPORE COUNT OF COMPRESSED FOOD BARS

(organisms per gram)

	Packaging and Three-Month Storage Condition				
Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
Beef, low moisture	0	0	0	0	0
Beef, high moisture	13	0	0	0	0
Beef, w/sorbitol	0	0	0	0	0
Beef, w/mono-, diglycerides	0	0	0	0	0
Dates, low moisture	5	0	0	2	0
Dates, high moisture	38	28	0	4	0
Dates, w/acetyl glyceride coating	3	0	0	0	0
Peas, low moisture	9	0	0	0	0
Peas, high moisture	0	0	0	0	0
Cereal, low moisture	5	0	0	0	0
Cereal, high moisture	4	0	8	0	0
Nonfat milk solids	55	94	44	42	82
Potato chips	0	0	0	0	0

TABLE A-XXIV

EFFECT OF THREE-MONTHS' STORAGE ON OXYGEN UPTAKE BY COMPRESSED FOOD BARS

(total milliliters of oxygen uptake per gram of dry matter, 10-day period)

Food Bar	Fresh	Air (100°F)	Vacuum (100°F)	Air (0-40°F)	Air (40% R.H., 70°F)
		Packaging and Three-Month Storage Condition			
Beef, low moisture	2,174	200	276	505	639
Beef, high moisture	2,282	397	536	704	680
Beef, w/sorbitol	763	245	269	415	624
Beef, w/mono, diglycerides	793	220	285	393	675
Dates, low moisture	106	265	319	181	160
Dates, high moisture	193	-	-	275	308
Dates, w/acetyl glyceride coating	710	252	294	-	-
Peas, low moisture*	580	452	568	578	482
Peas, high moisture*	510	515	546	576	638
Cereal, low moisture**	257	187	197	173	276
Cereal, high moisture**	409	251	317	326	256
Nonfat milk solids	281	288	288	278	232
Potato chips	254	209	261	204	226

* Based on average uptake over an eight-day experimental period.

** Based on average uptake over a seven-day experimental period.

APPENDIX B

FOOD BAR INGREDIENTS

Freeze-dried cooked ground beef, purchased from Wilson and Co., Inc., Prudential Plaza, Chicago 1, Ill. Product to conform to specifications cited in Quartermaster Corps, LP/P DES C-182-62, 20 June 1962.

Dehydrated chicken broth, purchased from Hennington, Inc., New York, N.Y.

Sorbitol, manufactured by Atlas Chemical Ind., Inc., Wilmington, Del.

Mono- and diglycerides, Atmul 84, manufactured by Atlas Chemical Ind., Inc., Wilmington, Del.

Pitted Sair dates, purchased from S. W. Noggle Co., 436 West 5th St., Kansas City, Mo.

Acetyl glycerides, Myvacet 9-40 and Myvacet 5-00, manufactured by Distillation Products, Inc., Rochester, N.Y.

Cooked, freeze-dried peas, purchased from California Vegetable Concentrates, Inc., 705 Whitmore Ave., Modesto, Calif.

Cornflakes, manufactured by W. K. Kellogg Co., Battle Creek, Mich.

Shortening, coconut butter flake, purchased from S. W. Noggle Co., 436 West 5th St., Kansas City, Mo.

Sugar, manufactured by California and Hawaiian Refining Corp., San Francisco, Calif.

Nonfat milk solids, purchased from Kraft Foods, Inc., 1504 Burlington Ave., Kansas City, Mo.

Potato chips, purchased from Kitty Clover Potato Chip Co., 817 Westport Rd., Kansas City, Mo.

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13. ABSTRACT

Objective methods are described for determining specific physical, chemical and microbiological properties of compressed food bars. The suitability of these methods was demonstrated by application to fresh and aged (3 months at $100^{o}F$) bars prepared from meat, fruit, cereals, vegetables, and dairy products and which represented broad concentration ranges of moisture, fat, protein, carbohydrate, and common approved chemical additives.

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
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Testing	8					
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